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# **THE EFFECT OF STRESS CRACK CORROSION AFTER USING DIFFERENT PWHT TEMPERATURES ON P91 STEEL**

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## Abstract

This work focuses on the heat treatment of welded joints and was developed in a partnership with the company TRATERME - Tratamentos Térmicos, L.<sup>da</sup>.

The importance of the heat treatment is affecting the microstructure to improve the performance of the material after welding. The crystal structure is rearranged specifically by changing temperatures. The importance of this rearrangement is to block the ferrite formation. First of all, this study analyses the effect of 4 different post welding heat treatment temperatures on P91, which is the abbreviation of the X10CrMoVNb9-1 steel. For this process, a shielded metal arc welding method with butt joining was used, which was necessary for the material not to overlap.

The entire process was made according to the standards ASME B31.1 and ASME B31.3.

During sample preparation, all the parameters were the same for all the specimens, except temperature; the temperatures were 730°C, 650°C, 800°C and 850°C. These selected temperatures were selected from the isopleth of the ternary Fe-C-Cr phase diagram for 9% (wt.) Cr. [2]

In the second part of the work, destructive, non-destructive, and mechanical tests were performed. These tests were necessary to prove the effectiveness of the PWHT on the material using stress crack corrosion and non-destructive tests.

First part of the results are related with mechanical tests. Different hardness values were obtained with a bench equipment. For stress crack corrosion test, three different types of non-destructive tests were needed for monitoring the defects on the corroded area. These results are contribute to the advance of this field of study, in the future.

**Keywords:** X10CrMoVNb9-1 steel, P91 steel, welded joint, preheat temperature, PWHT, SCC.

## Resumo

Este trabalho foca o tratamento térmico de juntas soldadas e foi desenvolvido em parceria com a , empresa TRATERME - Tratamentos Térmicos, L. <sup>da</sup>.

A importância do tratamento térmico é devido ao processo de soldadura, que poderá não resultar num bom desempenho do componente. Em primeiro lugar, este estudo analisa o efeito do tratamento térmico pós soldadura, realizado a quatro diferentes temperaturas, em P91, que é a abreviação do aço X10CrMoVNb9-1. Durante a preparação da amostra, todos os parâmetros eram idênticos em todos os espécimens, excepto a temperatura. As temperaturas são 730°C, 650°C, 800°C e 850°C respectivamente, para o espécime-tipo. Estas temperaturas foram seleccionadas da análise da secção vertical do diagrama-fase para %Cr=9. Como método de soldadura, foi usado soldadura de topo, de acordo com as normas ASME B31.1 e ASME B31.3.

Na segunda parte do trabalho prático, foram executados testes destrutivos, não-destrutivos, mecânicos e químicos. Estes testes foram necessários para testar a eficácia dos testes PWHT e SCC.

A primeira parte dos resultados está relacionada com ensaios mecânicos. Para o teste de corrosão sob tensão, três diferentes tipos de testes NDT forma necessários para monitorizar os defeitos na área corroída. Os resultados afiguram-se promissores para o futuro.

**Palavras-chave:** Aço X10CrMoVNb9-1, aço P91, junta soldada, temperatura de préaquecimento, tratamento térmico após soldadura, microestrutura, PWHT, SCC.

## Özet

Bu çalışma, sıcaklığın kaynaktan sonra yapılan ısıl işlem üzerindeki etkilerine odaklanmış olup TRATERME şirketiyle ortaklaşa gerçekleştirilmiş ve geliştirilmiştir.

Kaynak için ısıl işlem daha iyi bir performans sağlaması için önemlidir. Öncelikle bu çalışmada, X10CrMoVNb9-1 çeliği, kısaltması P91, için 4 farklı kaynak sonrası ısıl işlem sıcaklığı seçilmiştir. Malzemenin hazırlanması sırasında, bütün parametreler bütün malzemeler için aynı olup, sadece sıcaklık değiştirilmiştir. Bu sıcaklıklar numune ve deney sırası ile 730 °C, 650 °C, 800 °C, ve 850 °C olarak belirlenmiştir. Belirlenen bu sıcaklıklar için P91 çeliğinin faz diyagramının karbon yüzdesi sabit olarak alınan kesitinden yararlanılmıştır. Kaynak işlemi ve kaynak sonrası ısıl işlem sıcaklıklar için ASME B31.1 ve ASME B31.3 standartlarından faydalanılmıştır.

Çalışmanın ikinci kısmı uygulamalı olarak gerçekleştirilmiştir. Tahribatlı, tahribatsız numune ve mekanik testler uygulanmıştır. Bu testler, kaynak sonrası uygulanan ısıl işlemin ve stress kaynaklı korozyon testlerinin etkilerini ispatlamak açısından oldukça gereklidir. Aynı numune için uygulanan testler ve alınan sonuçlar yapılan testlerin farklılıkları ortaya çıkmasına yardımcı olmuştur.

Sonuçların ilk kısmında, sonuçlar mekanik testlerle ilgilidir. Farklı sertlik ölçüm cihazları ile birbirinden farklı sonuçlar elde edilmiştir. Sertlik testlerinden en iyi sonucu almak için ortamın tüm sarsıntılardan ve farklı dalga boyları yaratacak etkilerden uzak olması gerekmektedir, çünkü bu durum elde edilen verilerin doğruluğunu daha kolay ispatlanmasına yardımcı olacaktır. Stres kaynaklı oluşan korozyon testinde, numunenin farklı alanları için üç farklı tahribatsız deney yöntemine ihtiyaç duyulmuş olup, sonuçlar toplanan veriler ışığında değerlendirilmiştir. Sonuçlar gelecek deneyler için umut vericidir.

**Anahtar Kelimeler:** X10CrMoVNb9-1 çeliği, P91 çeliği, kaynak sonrası ısıl işlem, stres kaynaklı korozyon, mikrostrüktür, kaynak öncesi ısıl işlem.

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## List of Contents

1. Introduction.....	1
2. Literature Review.....	2
2.1. Material Description.....	2
2.1.1. Effect of the alloying elements.....	3
2.1.2. Continuous cooling transformation (CCT) diagram.....	5
2.2. Welding Process.....	7
2.2.1. Shielded metal arc welding (SMAW).....	7
2.3. Microstructural Zones (Base Metal, HAZ and Welding Zone).....	8
2.4. Heat Treatment of P91 Steel.....	10
2.4.1. Bake-out.....	10
2.4.2. Preheating and interpass heating.....	11
2.4.3. Post heating.....	11
2.4.4. Post weld heat treatment (PWHT).....	12
2.5. Stress crack corrosion (SCC).....	14
3. Experimental Work.....	16
3.1. Chemical composition of P91 pipe and welding consumable used in the investigation	16
3.2. Experimental procedure.....	16
3.2.1. Sample Preparation.....	16
3.3. Metallographic Sample Preparation.....	19
3.4. Hardness Measurements.....	19
3.5. Non-Destructive Tests.....	20
3.5.1. Digital radiography tests of welds.....	20
3.5.2. Dye Penetrant Test.....	21
3.5.3. Magnetic Particle Inspection Test.....	21
3.6. Stress Crack Corrosion Test.....	23
4. Experimental Results and Discussions.....	25
4.1. Metallographic Analysis Results.....	25
4.2. Hardness Measurements Results.....	27
4.3. SCC Test Results.....	29
5. Conclusion.....	37
6. References.....	39



## List of Figures and Tables

<b>Figure 1:</b> Continuous Cooling Transformation Diagram for P91 steel [2].....	6
<b>Figure 2:</b> Scheme of Shielded Metal Arc Welding circuit [9].....	8
<b>Figure 3:</b> Schematic representation of different heat affected zones [12].....	9
<b>Figure 4:</b> SCC variables.....	15
<b>Figure 5:</b> Assembly of preheating and interpass heating system.....	17
<b>Figure 6:</b> VAI 45 power generator of the induction heat treatment equipment.....	17
<b>Figure 7:</b> Welding Procedure of P91 steel.....	18
<b>Figure 8:</b> Graphic representation of the 5 hardness measuring zones.....	20
<b>Figure 9:</b> Assembly of the SCC experiment.....	24
<b>Figure 10:</b> Microstructure of P91 steel before PWHT, etching with Vilella's reagent.....	25
<b>Figure 11:</b> Microstructures of P91 steel base material after PWHT: 1- at 650°C; 2- at 730°C; 3- at 800°C; and 4- at 850°C, 500x, etching with Vilella's reagent.....	26
<b>Figure 12:</b> Microstructures of P91 steel welding zone after PWHT: 1- at 650°C; 2- at 730°C; 3- at 800°C; and 4- at 850°C, 500x, etching with Vilella's reagent.....	26
<b>Figure 13:</b> Microstructures of P91 steel HAZ and welding zone after PWHT: 1- at 650°C; 2- at 730°C; 3- at 800°C; and 4- at 850°C, 500x, etching with Vilella's reagent.....	27
<b>Figure 14:</b> Hardness values of the specimens before and after PWHT.....	28
<b>Figure 15:</b> Liquid penetrant test after PWHT.....	29
<b>Figure 16:</b> Liquid penetrant test after cutting the pipes: A. Outer surface; B. Inner surface...	30
<b>Figure 17:</b> The results of Radiation Test of P91 steel samples, before SCC test.....	30
<b>Figure 18:</b> MPI test of specimen 1 before the SCC test.....	31
<b>Figure 19:</b> Specimens before SCC tests.....	32
<b>Figure 20:</b> Weight change of P91 steel during SCC test.....	33
<b>Figure 21:</b> SCC test specimens during 6 weeks (A: week 1, B: week 2, C: week 3, D: week 4, E: week 5, F: week 6).....	34
<b>Figure 22:</b> Radiation test results of SCC test samples after 6 weeks.....	35

<b>Figure 23: MPI test of specimens after SCC experiment.....</b>	<b>35</b>
<b>Table 1: ASTM specification of P91 steel [1].....</b>	<b>2</b>
<b>Table 2: International specifications of 9%Cr-1Mo steel [2].....</b>	<b>2</b>
<b>Table 3: Typical chemical compositions of recent variants of P91 steel [4].....</b>	<b>3</b>
<b>Table 4: Chemical composition (wt %) of the P91 pipe used in the investigation.....</b>	<b>16</b>
<b>Table 5: Chemical composition (wt %) of the welding consumable [26].....</b>	<b>16</b>
<b>Table 6: Processing parameters of PWHT for P91 steel.....</b>	<b>18</b>
<b>Table 7: Chemical composition of Vilella's reagent [28].....</b>	<b>19</b>
<b>Table 8: Hardness values for P91 steel from the supplier.....</b>	<b>28</b>
<b>Table 9: Weight changes of P91 steel samples during SCC test.....</b>	<b>32</b>

## List of Symbols and Abbreviations

AC: Alternating Current.

$A_{CM}$ : Upper critical temperature.

$A_{C1}$ : The temperature at which austenite begins to form on heating.

$A_{C3}$ : In hypoeutectoid steel, the temperature at which transformation of ferrite into austenite is completed upon heating.

ANSI: American National Standards Institute.

ASME: American Society of Mechanical Engineers.

ASTM: American Society for Testing and Materials.

atm: atmosphere.

BCC: Body Centered Cubic.

BS: British Standard.

CCT: Continuous Cooling Transformation.

DC: Direct Current.

FCC: Face Centered Cubic.

g: gram.

GCHAZ: Grain Coarsened Heat Affected Zone.

GRHAZ: Grain Refined Heat Affected Zone.

GTAW: Gas Tungsten Arc Welding.

HAZ: Heat Affected Zone.

HV: Hardness Vickers.

ISO: International Organization for Standardization.

$K_{ISCC}$ : Stress Intensity Factor.

kg: Kilogram.

kHz: Kilohertz.

mL: milliliter.

mm: millimeter.

MPa: Megapascal.

MPI: Magnetic Particle Inspection.

NDT: Non-Destructive Testing.

P: Pipe.

PWHT: Post Welding Heat Treatment.

SCC: Stress Crack Corrosion.

SMAW: Shielded Metal Arc Welding.

UV: Ultraviolet.

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## 1. Introduction

Ferritic martensitic 9-12% Cr steels have been developed worldwide over the last two decades for elevated temperature, nuclear and fossil energy applications. P91 steel is commonly used for high temperature applications up to 650°C [1].

In this study, P91 ferritic martensitic steel pipe was used. This is a modified creep resisting steel that presents higher stress values than its previous version (P9). Besides the major alloying elements such as carbon, chromium, silicon, molybdenum, other micro alloying elements, e.g. vanadium, niobium and nitrogen, improve the properties of the P91 steel. The major application areas of this steel are power generating and process industries for components such as boilers, headers, superheaters, due to its elevated thermal properties.

Mechanical properties of P91 steel are similar compared with the previous grades, yield strength of 415 MPa (minimum), tensile strength of 585 MPa (minimum), and the mean Vickers hardness values of HV10 250 at room temperature [2].

Mostly, P91 microstructure is obtained by normalizing and tempering heat treatments. The normalizing temperature range is between 1040°C and 1080°C and the tempering temperature range is from 750°C to 780°C [3]. After tempering, the microstructure of the material is a mixture of tempered martensite with carbide precipitates. Due to precipitation hardening, the carbide precipitates improve the creep rupture strength of the material.

Despite the fact that P91 steel has high strength, it has been found that the mechanical properties of the heat affected zone (HAZ) region of weld joints of P91 steel are lower compared to both base material and welding area. During high temperature service, lower mechanical properties create cracking problem in HAZ regions.

Creep resistant P91 steel is a high strength alloy that transforms entirely to martensitic structure during air cooling. Main alloying elements of P91 steel, carbon, molybdenum, and chromium, decrease the  $M_s$  temperature and improve its hardenability.

During welding process, it is impossible to avoid hydrogen formation and the risk of hydrogen induced cracking. To avoid this crack formation, after welding process, post welding heat treatment is a very critical process.

This thesis focuses on the effect of the stress crack corrosion after post welding heat treatment process. Microstructural analysis, hardness measurements, and non-destructive techniques were performed before and after heat treatment in base material, heat affected zones and welding area.

## 2. Literature Review

### 2.1. Material Description

The steel X10CrMoVNb9-1 family, designed by ASTM A335/A 335M - 06, is used in high temperature applications such as boilers, steam pipes, heaters, etc. The ASTM A335 family, known as seamless martensitic alloy-steel pipe for high-temperature service, evolves into the P91 grade (P being the abbreviation of pipe), the material with the highest creep and oxidation resistance properties [3].

During this evolution, the main difference resides in their chemical composition, with particular emphasis to the chromium (Cr) content of each grade. Thus, developments and researches on this material are going on to increase the service temperatures up to 650°C and higher. Due to this evolution, this alloy was incorporated in an ASTM specification to design P91 steel. This specification is presented in table 1.

Table 1: ASTM specification of P91 steel [4]

Grade	Specification	Composition (%)							
		C	Mn	P	S	Si	Cr	Mo	V
P91, T91	A199, A200,	0.08-	0.3-	0.020	0.010	0.20-	8.00-	0.85-	0.18-
	A213, A335	0.12	0.6			0.50	9.50	1.05	0.25

P91 steel has been manufactured all around the world and it has different variety of international standards as shown in table 2.

Table 2: International specifications of 9%Cr-1Mo steel [5]

Country	Standard	Grade
USA	ASTM A213	T91 (seamless tubing)
USA	ASTM A335	P91 (seamless pipe)
UK	BS 1503	Gr 91 (forging)

Germany	DIN 17175	X10 CrMoVNb 9 1
Japan		HCM-9S
France	NF A 49213	T U Z 10CDVNb 09-01

In ASME code 31.1, P91 steel is supplied in normalized and tempered condition. According to this code, minimum normalizing temperature is 1050°C and minimum tempering temperature is 730°C. Before normalizing we should obtain a homogeneous austenitic structure; air cooling transforms austenite to martensite.

#### 2.1.1. Effect of the alloying elements

The large Cr and Mo contents (table 1) and micro alloying elements such as V and Nb improves the high temperature properties of P91 steel. Despite the low carbon concentration, it promotes the hardenability and strength in martensitic steels. For P91 steel, expected hardness values are between 200 and 263 HV [3]. Hardness increase occurs through solution hardening by alloying elements such as Mn, Si, Cr, Mo and Nb.

Since the creation of P91 steel, many studies were developed on the effects of the alloying elements. In the meantime, using ASTM specifications as a main alloying structure, scientists are developing the varieties of creep resistant steels. Changing the compositions of alloying elements complement the strength at elevated temperatures. The recent variants of P91 steel are presented in table 3.

Table 3: Typical chemical compositions of recent variants of P91 steel [6]

Elements	Chemical Composition (wt. %)			
	ASME specifications			
	T/P9	T/P91	T/P92	T/P911
Carbon, C	Max. 0.15	0.10	0.124	0.105
Silicon, Si	0.20-0.65	0.38	0.02	0.2
Manganese, Mn	0.80-1.30	0.46	0.47	0.35
Phosphorous, P	Max. 0.030	0.020	0.011	0.007
Sulphur, S	Max. 0.030	0.002	0.006	0.003
Chromium, Cr	8.5-10.5	8.10	9.07	9.16



Molybdenum, Mo	1.70-2.30	0.92	0.46	1.01
Vanadium, V	0.20-0.40	0.18	0.19	0.23
Niobium, Nb	0.30-0.45	0.073	0.063	0.068
Tungsten, W	-	-	1.78	1.00
Boron, B	-	-	0.003	-
Nitrogen, N	-	0.049	0.043	0.072
Nickel, Ni	Max. 0.30	0.33	0.06	0.07
Aluminum, Al	-	0.034	0.002	-

The main alloying elements for this family are carbon (C), chromium (Cr), molybdenum (Mo), nickel (Ni) and vanadium (V). The addition of these alloying elements is usually done in order to increase hardness, strength and corrosion resistance of the product [6].

P91 (also known as modified 9Cr-1Mo) is extensively used in power plants due to its creep and corrosion properties at high temperatures. The unique martensitic microstructure of P91 ensures favorable strength and toughness for high temperature applications. Also post welding heat treatment is the most important and critical step in the fabrication of P91 for obtaining the desired in service performance.

P91 is a martensitic chromium-molybdenum steel containing tungsten, vanadium, niobium and nitrogen as micro-alloying elements. The role of each element in the alloy is as follows:

- Carbon is the basic alloying element of all steels. It provides high hardness and tensile strength in carbon low alloys. However, if the steel's carbon content is higher than standard range, the material's weldability decreases.
- Chromium is the main alloying element of P91 steel. Cr mainly improves the high temperature resistance, and also oxidation and corrosion resistance of the P91 steel. Cr is a carbide former and stabilizer. In P91, the oxidation and corrosion resisting effects are significant at elevated temperatures.
- Molybdenum acts as ferrite stabilizer. Likewise, it increases high temperature strength and corrosion resistance of P91 steel, and contributes to the hardenability and toughness of the alloy.

- Vanadium forms carbide and nitride precipitates which results in finer grains. When vanadium forms a compound with nitrogen, this reaction creates VN compound that improves the strengthening properties of the P91 steel.

- Nickel enhances corrosion resistance, high toughness, and high temperature strength of the alloy. It inhibits the  $\delta$ -ferrite formation, and decreases  $A_1$  transformation temperature.

P91 steel presents better creep, tensile and fatigue properties compared with the grades in the alloy family, being able to resist high pressures (between 170 and 230 bar) and high temperatures (between 500°C and 600°C).

Some of the key advantages of using P91 steel are [1, 5]:

- High creep strength;
- High ductility;
- Good weldability, and the possibility of easy bonding to other martensitic steels;
- Good thermal conductivity and low thermal expansion coefficient;
- Good anticorrosive properties and resistance to aqueous and gaseous environments, including hydrogen-rich environments;
- Ease of machining.

Due to these properties, P91 is the best material for the high temperature applications such as boiler superheaters, tube heaters, headers, and duct pipes in infrastructures such as conventional and nuclear plants, and petrochemical installations.

#### 2.1.2. Continuous Cooling Transformation (CCT) diagram

The isothermal transformation diagram of steel is an important tool for design and development of processes such as welding and heat treatment. In experiments, constant cooling rates are usually used. CCT diagrams display the phase transformation behaviour as a function of cooling rate. It helps to predict the final microstructure and properties of the material which are resulting from cooling rate. During this transformation, cooling rate affects the final microstructure. From austenite to pearlite transformation, austenite transforms over a wide range of temperatures and time; a specimen will have mixed microstructure at the end of the transformation. An isothermal transformation diagram shows that the time and temperature relationships are applicable to transformation accomplished at constant temperatures but not applicable to heat treatments involving transformation under continuous cooling conditions. Usually, P91 steel is heated to the austenite region and cooled to room temperature gradually at different cooling rates. Cooling rates depend on type of treatment, size and the shape of the

piece [2]. CCT diagrams are widely used to estimate the microstructure and mechanical properties after continuously cooling from austenitizing temperature.

For P91 steel, to achieve desired properties, such as hardness, microstructure, normalizing and tempering treatments are vital. In the normalization process, the steel is heated to, and kept at temperatures approximately 55°C above the upper critical temperature ( $A_{c3}$ ) to complete austenitic transformation [7]. When the temperature is uniform, it is allowed to cool to room temperature in air in order to maintain the homogeneity. Once enough time has elapsed for the material to achieve a uniform temperature and for homogenization process to take place, the material is left to cool to room temperature. When the material reaches the room temperature, it has passed the two most important temperatures, the martensitic phase start and finishing transformation temperatures [7, 8]. These temperatures are represented in the continuous cooling transformation (CCT) diagram in figure 1.

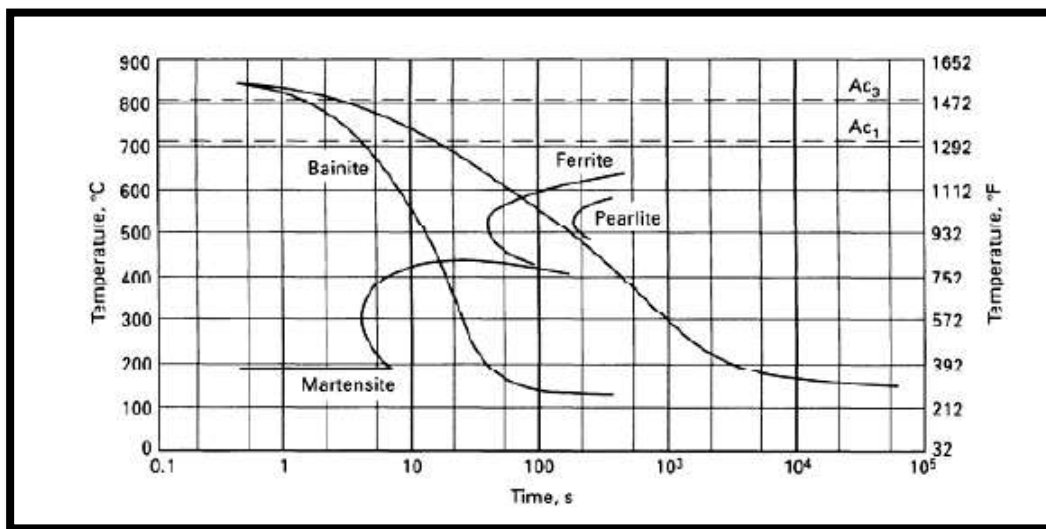


Figure 1: Continuous Cooling Transformation diagram for P91 steel [7]

Martensite is a metastable microstructure. The hardening treatment of P91 steel comprises heating the material to  $A_{c3}$  temperature to provide a fully austenitic structure and then normalized to form martensitic structure. If P91 is not cooled rapidly, some retained austenite may remain in the microstructure, which can cause undesirable properties such as susceptibility to cracking, low ductility and toughness, after post welding heat treatment process.

Due to the high hardenability of P91 steel, the alloy will have to be tempered, a common treatment for this material in order to achieve high hardness, minimum distortion and low

cracking tendency. Tempering will relieve the residual stresses accumulated during cooling from normalizing temperatures, and will also contribute towards the increase of the material's ductility and toughness. Time, temperature, and cooling rate control have vital importance in the tempering process. Tempering is performed between 175 and 705°C to gain toughness and soften the metal [7]. The material is then air-cooled down to room temperature in order to obtain desired mechanical properties such as high toughness, hardness and ductility and also to use for stress relief.

The main reasons for conducting the normalizing and tempering heat treatment processes to the P91 steel are [5]:

- Reduce the high strength of the weld metal;
- Eliminate the likelihood of cold crack failure;
- Achieve residual stresses reduction.

## 2.2. Welding Process

### 2.2.1. Shielded Metal Arc Welding (SMAW)

The shielded metal arc welding process is the most widely used welding process in the world. This process is preferable due to its easiness, and low investment and equipment cost. Nevertheless, it is one of the most difficult one in terms of required training and experience to achieve the sought results, since SMAW is a fully manual process.

In this process, materials are bonded when heat from an arc is stabilized between the point of a welding consumable covered wire and the workpiece of the metal joint to be welded. The heat of the arc is used for melting the base metal and the tip of the welding electrode. The electric circuit and its components can be seen in figure 2. When in operation, one of the cables is connected to the work lead, whilst the other is connected to the electrode holder. Welding starts when the electric arc is creating a contact between the tip of the electrode and the work lead. The concentrated heat of the arc melts the tip of the electrode, and then transfers through the arc into the weld pool. The arc must be moved on the work lead at constant speed, ensuring a proper arc length is maintained. The arc can reach a temperature up to 5000°C, and melting of the base material will occur shortly after arc initiation [9].

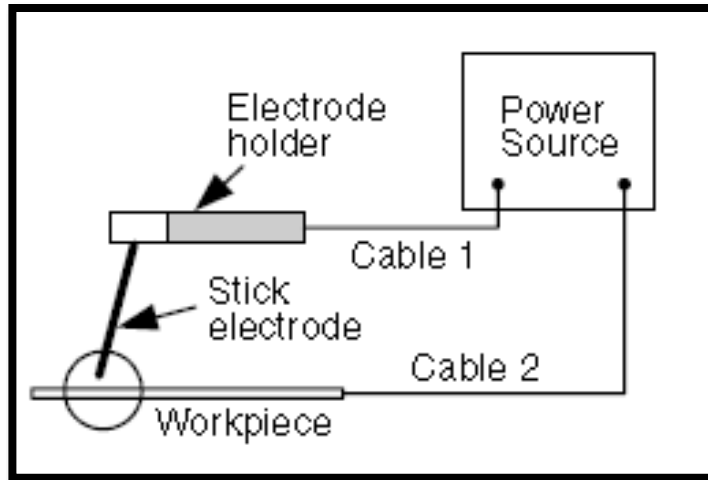


Figure 2: Scheme of Shielded Metal Arc Welding circuit [10]

In the SMAW process, both alternating (AC) and direct (DC) currents can be used, although DC is most commonly used over AC currents. The current only flows in one direction and it brings advantages over alternating current, such as better control of parameters, less sticking, and easy arc starting. The electrode cable is connected to the positive pole, whilst the workpiece is connected to the negative output, thus creating the reverse polarity connection most commonly used in this process. This type of connection brings good penetration and bead profile [10, 11].

The advantages of SMAW method are [10, 11]:

- The equipment is easy to use, cheap and portable;
- No need for another filler metal;
- No need for another gas or flux;
- Useful for complex shaped geometries;
- Suitable for most common metals and alloys;
- Allows welding in any orientation.

SMAW welding is widely used in applications such as naval industry, pipelines, offshore platforms, and steel constructions. Also this method is suitable for welding low and high alloy steels, stainless steels, and cast iron.

### 2.3. Microstructural zones (Base Metal, HAZ and Welding Zone)

The zones whose microstructures are affected as a result of welding procedures includes the base material, the weld metal and the heat affected zone (HAZ).

Base metal is the material to be welded. In this region, base material is not affected by the welding procedure, except the part close to the fusion boundary. Thus, due to the welding process, the base material is in the circumstance of residual, transverse and longitudinal stress.

Heat Affected Zone (HAZ) is a part of base material which has not been melted during welding process. In addition to that, the microstructure and mechanical properties have been adjusted. During welding, HAZ area's microstructure has been changed by the heat as a consequence of thermal cycle. Due to the thermal conditions and distance from the fusion line, the HAZ is divided into three microstructural areas, which are grain refined heat affected zone (GRHAZ), grain coarsened heat affected zone (GCHAZ), and intercritical heat affected zone. The importance of these areas are related to its propensity to failures.

Weld material zone involves a mixture of filler metal, which comes from welding consumable, and base metal. In this zone, filler and base metal fully melted and creates homogeneous structure [5].

These zones are represented in figure 3.

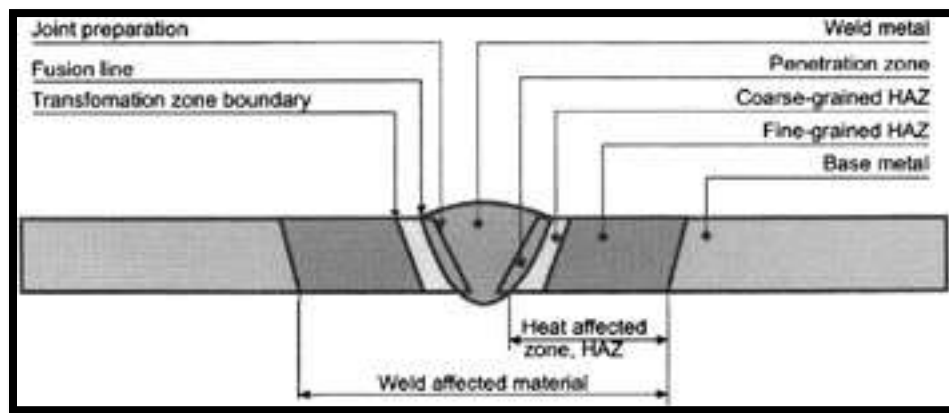


Figure 3: Schematic representation of different heat affected zones after welding [12]

#### 2.4. Heat Treatment of P91 Steel

Each step of the heat treatment processes has different importance. Application of heat treatment with precise control of preheat, interpass, and post weld heat treatment operations are required to ensure that the most favorable mechanical and chemical properties are obtained. To avoid hydrogen cracking in this alloy, controlling preheat and interpass temperatures and post-baking operations is necessary. Flame, electrical resistance, electrical induction heating and furnace heating have been successfully used. Temperature monitoring

and control of thermal gradients are important. This is a reason that local flame heating is not recommended and should not be permitted. Changes in thickness, chimney, and position effects must also be considered as important factors for controlling thermal gradients.

#### 2.4.1. Bake-out

A post weld bake-out may be of critical importance, especially for heavy sections or when flux-type processes are used. The process involves maintaining the preheat/interpass temperature for an extended period of time subsequent to interruption or completion of the welding. To establish the required processing time, factors such as material thickness, duration of the exposure of the weld to the heat regime, and the extent of “low hydrogen” practices have to be considered. When suitable preheat is applied, and carefully selected consumables are used, bake-out step can be eliminated.

Bake-out is performed to remove excessive hydrogen from the material, formed during manufacturing, and processing. The main purpose of removing hydrogen is to avoid hydrogen induced cracking in the welded area or heat affected zone. However, when hydrogen is present, care must be taken so that the hydrogen partial pressure does not result in material cracking. For this reason, temperature limitations can be imposed when conducting bake-out [13].

Since the purpose is to promote diffusion to free surfaces, time and temperature should be carefully selected in accordance with the hydrogen mobility, diffusion, initial and desired hydrogen content, and temperature restrictions of the material.

It is recommended that the bake-out process is conducted at 230-315°C for 2 to 4 hours. When specifying experienced based parameters, it is recommended that time be specified as a function of thickness in order to account for the variable diffusion path with minimum time requirement. Temperature restrictions may be needed to prevent high temperature hydrogen attack and adverse metallurgical reactions such as temper embrittlement [14].

#### 2.4.2. Preheating and interpass heating

A typical maximum interpass temperature is 300°C; lower temperatures are acceptable but higher temperatures should not exceed 370°C in order to achieve adequate mechanical properties in the heat affected zone and weld metal [14]. The maximum interpass temperature helps to prevent the possibility of hot cracking due to the silicon and niobium contents of the weld metal. Field operations rarely have problems with interpass temperature on heavy sections.

These heating processes normally have similar aims and are designed to achieve the minimum preheat and interpass temperatures. The former is instantaneously applied to the base material just before welding, and the latter to the weld area surpassing each pass in a multi-pass weld.

The first objective of this process is to restrict hydrogen cracking in the weld metal and heat affected zone. This goal is accomplished by the interaction of several parameters such as driving off moisture beyond the start of welding, decreasing the cooling rate, and increasing the rate of hydrogen diffusion. The second reason for conducting preheating and interpass heating is the redistribution of solidification stresses, which results in a slower processing rate. Lastly, the process also reduces the cooling rate in materials that form hard or brittle microstructural constituents when cooled too rapidly from welding temperatures.

The temperature requirements depend on the composition, time between passes, base material thickness, preheat temperature, environmental conditions, and heat input from welding. There are special fabrication codes, by ASME and ANSI, to make sure these requirements are fulfilled. Most of the welding procedures specify a minimum interpass/preheat temperature that must be maintained whenever welding takes place. Most of welding procedures also specify the maximum limit of interpass temperatures, which may not be surpassed prior to depositing the next pass in the same area. A maximum interpass temperature can be specified for metallurgical reasons such as maintaining the notch toughness of ferritic steels or corrosion resistance of austenitic stainless steels and some non-ferrous alloys. It is also specified for the health and effectiveness of the welder [14].

The literature suggests that 200°C is adequate for preheating P91 welds. Manufacturers typically aim for 200-250°C, but will go as low as 121°C for root and hot pass layers, thin-walled components, or when Gas Tungsten Arc Welding (GTAW) is utilized [15].

#### 2.4.3. Post Heating

This process is executed once all heating performed after welding has been stopped, and before the Post Welding Heat Treatment (PWHT) begins. Therefore, it is generally recognized that post heating is performed at lower temperature: 149-316°C versus 538-760°C for PWHT [14].

The first aim of post heating is the removal of hydrogen, thus preventing hydrogen induced cracking. That is also known as delayed cracking since it can occur up to 48 hours after the weld has been cooled to room temperature. Hydrogen induced cracking can occur when the introduction of hydrogen from the welding consumables or base material is not adequately



controlled, or when preheat/interpass is not sufficient. This failure mode is a special concern when joining high strength and alloyed steels.

If post heating is considered necessary the minimum preheat/interpass temperature should be maintained until the application of such post heating.

Usually, post heating is applied in situations where some delay is expected between the completion of welding and the application of PWHT and is not practical or cost effective to maintain the preheat/interpass temperature until PWHT. Likewise, a delay may be necessary before completion of welding. When a delay is not practical to maintain the interpass temperature during welding, intermediate post heating can be used.

For this process, the ASME standard has fabrication and repair codes, which are generally associated with temper bead or controlled deposition welding when used as an alternative to PWHT. It is recommended to specify time as a function of thickness to account for the variable diffusion path, with a minimum time requirement. Temperatures in the range of 260-316°C for 2 hours (minimum) per 25 mm of thickness are consistent with requirements for carbon and low alloy steels found in various codes. Nevertheless, based upon concerns with hydrogen trapping, it appears appropriate to use temperature of 316°C and higher. Temperature restrictions are necessary due to adverse metallurgical reasons [14, 15, 16].

#### 2.4.4. Post Weld Heat Treatment (PWHT)

Application of PWHT is absolutely necessary with Grade 91 welds, regardless of the pipe diameter or thickness. PWHT is one of the most important steps in obtaining satisfactory welds. The PWHT methodology and implementation must be verified to ensure that the welds are in fact receiving PWHT at the correct temperature. In addition, thermocouples or qualification testing will be necessary. To obtain reasonable levels of toughness, tempering of the martensitic microstructure will be essential [14, 16].

Post welding heat treatment is generally performed at a higher temperature and with different objectives than post heating. As with post heating, PWHT may need to be applied without allowing the temperature to decrease below the minimum for preheat/interpass.

Local PWHT of carbon and low alloy steels is typically performed below the critical transformation temperature. The critical transformation temperatures indicate where the microstructure of steel begins and finally completes a change from body centered cubic (BCC) to face centered cubic (FCC).

There are several reasons why local supercritical post welding heat treatment (such as annealing or normalizing) above the upper critical transformation temperature is not desirable. First of all, the temperature gradient fundamental to local PWHT would produce critical temperature regions. Depending upon the previous heat treatment of the material, this could result in a deleterious effect upon properties, such as tensile/yield strength, toughness, and local discontinuities. In addition, low material strength at supercritical temperatures increases the likelihood of distortion.

PWHT can have both beneficial and detrimental effects. Tempering, relaxation of residual stresses and hydrogen removal, are all benefits of PWHT. Ensuing benefits, such as avoidance of hydrogen induced cracking, dimensional stability, and ductility, will result in increased toughness and corrosion resistance. It is important for PWHT conditions to be determined based on the desired objectives.

Excessive or incorrect PWHT temperatures and long dwelling times may reduce tensile strength, creep strength and notch toughness. Notch toughness is mostly caused by embrittlement due to the precipitates formation. The influence of PWHT on properties generally depends on the composition of the weld metal and base metal, and previous thermal and mechanical processing of the base metal [17, 18].

The need for PWHT is consistently driven by direct requirements which are generally triggered by material type and thickness. In the standards, there are fabrication codes and these codes provide detailed requirements regarding local PWHT. PWHT is usually aimed at reducing susceptibility to brittle fracture and also is targeted to improve notch toughness and relaxation of residual stresses [15, 19].

## 2.5. Stress Crack Corrosion

Stress crack corrosion (SCC) occurs due to the combination of mechanical stresses in a corrosive environment. SCC can occur in a numerous ways, and it is a subtle form of corrosion. Likewise, it may happen unexpected and rapidly, and it will result in failure of the material or leakage. To initiate SCC, there are three requirements: a susceptible metal, a corrosive environment and sufficient tensile stress. This type of corrosion is very rare but its consequences are very expensive and destructive [20].

Austenitic stainless steels suffer from stress corrosion cracking mostly in hot solutions containing chloride-based compounds. The temperature should be above 70°C for the process

occur, though SCC can take place at lower temperatures in some cases, namely acid solutions. The failure continues at low stresses and normally happens as a result of residual stresses from welding or fabrication. The cracking is generally transgranular, though it transforms to an intergranular path as a result of steel sensitization [21].

Stress crack corrosion has three basic mechanisms. The first mechanism is active path dissolution. The typical active path is the grain boundary, where the segregated impurity elements can make the passivation harder. This process can initiate without stress; stress, however, will open a crack, and make diffusion easier, thus promoting corrosion medium spreading and faster material corrosion. The second mechanism is hydrogen embrittlement. Hydrogen can diffuse between other atoms faster and easily. The diffused hydrogen can cause material fracture; it can easily open a cleavage and create intense local plastic deformation. This deformation leads to embrittlement, and cracking will be either intergranular or transgranular. Hydrogen has low solubility but high diffusivity in BCC iron. On the other hand, in FCC iron, hydrogen has high solubility but low diffusivity. This means corrosion of P91 steel takes longer time compared with ferritic iron. The final property is film induced cleavage. In this mechanism, the walls of the material and the crack point are covered with a film. Plastic strain ruptures the film at the crack point, and the crack migrates from the film into the matrix. The mechanism usually causes a transgranular fracture [22, 23].

For the occurrence of stress crack corrosion, the following three parameters are essential (see Fig. 4); if one of them is missing, SCC may not take place.

As highlighted in Figure 4, in order to prevent stress crack corrosion, one of the variables needs to be removed. For example, applying post welding heat treatment will reduce residual stresses and keep them below critical value. Likewise, selecting less susceptible materials for the required applications will retard the corrosion process.

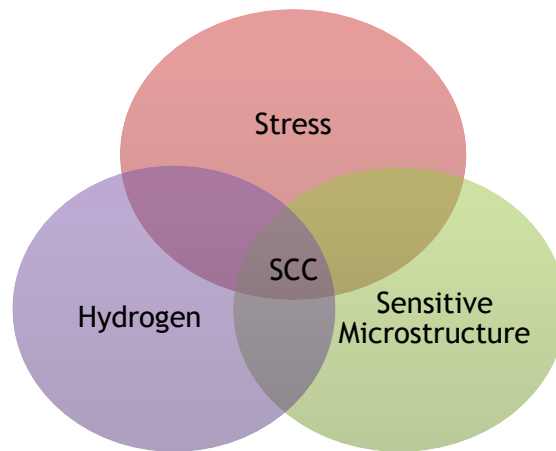


Figure 4: SCC variables

Design details, such as welds, pits, changes in section, etc., improve the stress concentration of the material. When the stress concentration of the material should pass the threshold, then SCC will take place by the effect of corrosive environment. In addition, stress can be produced as a result of the material service. For example, the material might be exposed to corrosive environment such as sea water, or a temporary operation that leaves corrosive residues on the material [21, 23].

### 3. Experimental Work

#### 3.1. Chemical composition of P91 pipe and welding consumable used in the investigation.

In this study, the material was P91 creep resisting steel manufactured by Amal. Table 4 shows the chemical composition of 5.08mm thick P91 pipe steel which was investigated in the current work. The P91 steel was supplied as normalized and tempered condition. The received P91 steel structure was tempered martensite and the mean hardness value was 235 HV.

Table 4: Chemical composition (wt %) of the P91 pipe used in the investigation

Composition (wt %)													
C	Mn	Si	Ni	Cr	Mo	V	P	S	Al	Ti	Nb	N	Zr
0.12	0.6	0.5	0.4	9.50	1.05	0.25	0.002	0.001	0.002	0.001	0.1	0.007	0.001

The welding consumable used were KESTRA under the classification of DIN 8575: E Cr Mo 2 B 26 and AWS 5. 5: E 9018- B3/B3 L. It has 3.25mm wire diameter for the root pass. In table 5, the chemical composition of the consumable is presented.

Table 5: Chemical composition (wt %) of the welding consumable [24]

Composition (wt %)								
C	Mn	Si	Ni	Cr	Mo	V	Nb	N
0.1	1.0	0.25	0.8	9.0	1.0	0.25	0.08	0.05

#### 3.2. Experimental procedure

Experiments were conducted in three stages: welding, post welding heat treatment at four different temperatures, and finally testing of all samples by using metallographic examination, hardness test, Non-Destructive Testing (NDT) and Stress Crack Corrosion (SCC) methods.

##### 3.2.1. Sample preparation

Samples preparation started by a 2 h bake-out process at 200°C, followed by an overnight cooling. Afterwards, thermocouple were connected to the pipes and previously controlled by the equipment in order to check connections (figure 5). After verifying the connections, the pipe was covered with a layer of induction resistant, refractory wool and finally by a blanket. The equipment was programmed, and preheating and interpass heating processes were started.

At the same time, electrodes for the welding were inserted in the stove for 1 h for hydrogen removal. The preheating and interpass heating time-temperature graph is presented in appendix 1.



Figure 5: Assembly of preheating and interpass heating system

Welding procedure was occurred at TRATERME. The welding process was carried out using SMAW method. During welding process, the thermal cycle was monitored using embedded thermocouples. The preheat temperature prior to welding was  $200^{\circ}\text{C}$ , and interpass temperature was controlled at  $250^{\circ}\text{C} \pm 10^{\circ}\text{C}$ .

For the PWHT process, the induction heating method was applied at the request of the company and the equipment VAI 45 was used. The heat treatment equipment is shown in figure 6. To initiate the process, the heating rate and temperatures were selected for all the specimens, as presented in table 6.



Figure 6: VAI 45 power generator of the induction heat treatment equipment

Table 6: Processing parameters of PWHT for P91 steel

Specimen Number	PWHT Parameters	Temperature (°C)
1	Lower than transformation temperature	650
2	Standard heat treatment temperature	730
3	Higher than actual PWHT but inside the same transformation temperature range	800
4	Higher than transformation temperature	850

After welding, all the specimens were heated to 250°C, at a heating rate of 200°C/h. They were held at those temperatures for 2 h, then cooled at the same cooling rate. The time and temperature profiles for each heat treatment are shown in appendixes 2, 3, 4 and 5. The welding procedure of all specimens is presented in figure 7. Post welding heat treatment was performed at 650°C, 730°C, 800°C and 850°C, respectively.

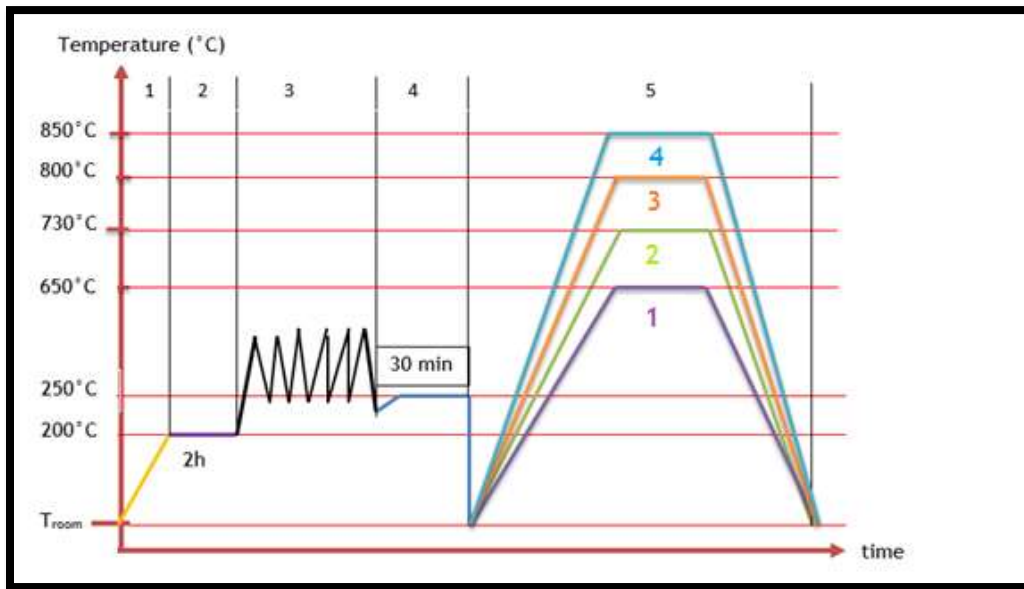


Figure 7: Welding procedure and PWHT of P91 steel

Once all the PWHTs were complete, all the specimens were subjected to dye penetrant inspections in order to detect the eventual presence of cracks. After collecting all data, the pipes were ground by abrasive discs and cut into 4 pieces of certain diameters by an abrasive cutter. Next, the samples were again inspected using a dye penetrant to check the

discontinuities of the inner surface of the pipes. Finally, after all samples were prepared, destructive, non-destructive, and mechanical tests were conducted before and after SCC test.

### 3.3. Metallographic Sample Preparation

Characterization of the heat treated samples was implemented using optical microscopy. Optical microscopy was used to detect general microstructural features.

The specimens for optical microscopic examination were longitudinal sectioned. All samples were hot mounted by using phenolic mounting resin. In addition, all the specimens were prepared according to the standard metallographic sample preparation procedure ASTM E3-11 [25]. The samples were then ground by using 180, 220, 320, 400, 600, 800, 1000 and 4000 grit SiC grinding paper with water as a coolant. For polishing step, a napped cloth with addition of an aqueous-based colloidal silica suspension and diamond paste was used.

The microstructural features were revealed by etching with Vilella's reagent, which consists of picric acid, hydrochloric acid and ethanol (table 7). Vilella's reagent allows to observe general structure, grain contrast better than the other etchants for heat treated steels. It etches the grain boundaries where carbides are located.

Table 7: Chemical composition of Vilella's reagent [26]

Chemical Composition	Etchant Application
1g Picric acid	<ul style="list-style-type: none"><li>• Dip etching for 30 seconds.</li><li>• Rinse in alcohol and then dry.</li></ul>
5 mL concentrated HCl	
95 mL Ethyl alcohol	

### 3.4. Hardness measurements

The Vickers hardness test is mostly used for small sections. The test procedure, ASTM E-384, specifies the load to be used along a diamond indenter, to make an indentation which is measured and converted to a hardness value [27]. This method is convenient for testing a wide range of metals, ceramics, composites, and other materials, as long as test samples are carefully prepared. A square-base-pyramid-shaped diamond indenter is used for the Vickers tests. Typically loads are very small, ranging from a few grams to several kilograms, although "macro" Vickers loads can range up to 30 kg or more.



Macrohardness measurements were implemented by using a Vickers hardness testing machine for hardness profiles of the welding, HAZ and base material before and after post welding heat treatment process.

Vickers hardness measurements were performed with a 10 kg load using the Struers hardness device on each heat treated sample. Recorded hardness values are the average of 5 measurements of the welding, HAZ and base material area. Figure 8 shows the hardness measurement points on the pipe. The hardness measurements were obtained on samples mounted in bakelite then polished and etched by Vilella's reagent.

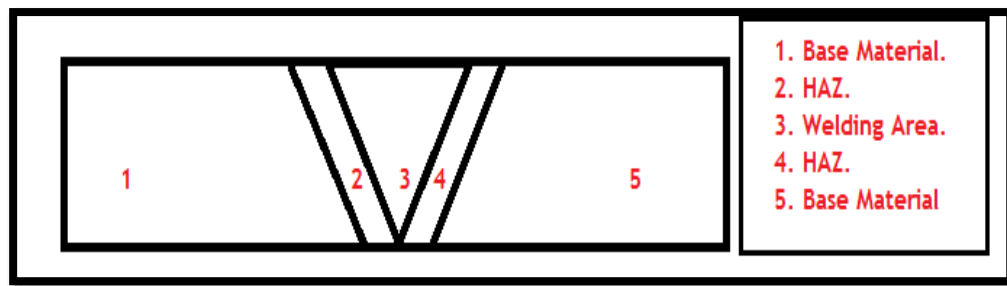


Figure 8: Graphic representation of the 5 hardness measuring zones

### 3.5. Non-Destructive Tests

#### 3.5.1. Digital radiography tests of welds

Radiography is one of the most well-known Non-Destructive Tests (NDT). Radiography can be used to collect permanent image of surface discontinuities. The same discontinuities can be re-measured after a certain period of service life and the results can be compared to measure the variation of the discontinuity. The main advantage of this method is to provide more information about the base material, HAZ and welding area's microstructure without destroying the component.

Radiography test was performed under the guidance of the standards: ASTM E-94-04: Standard Guide for Radiographic Testing, E1030-05: Standard Test Method for Radiographic Examination of Metallic Castings and E-1416-96: Standard Test Method for Radioscopic Examination of Weldments [28, 29, 30].

Before starting the examination, all the specimens were cleaned, and fixed on the screen. After that, the room has to be dark to achieve better quality image. The direction of radiation is governed by the geometry of the specimen, the radiographic coverage, and quality

requirements stipulated by the applicable job order or contract. Whenever practicable, the central beam of the radiation should be placed perpendicular to the surface of the film. The standard ASTM E94-04 provides examples of preferred source and film orientations and examples of specimen geometries and configurations on which radiography is impractical or very difficult [28].

### 3.5.2. Dye Penetrant Test

It is a widely applied and low cost inspection method used to locate visible surface defects in all non-porous materials. The penetrant is applied to all non-ferrous and ferrous metals. This test is used to detect welding surface defects such as cracks, surface porosity, and leaks on the surface.

The dye penetrant can be applied to the specimen by dipping, spraying, or brushing. Each dye penetrant test requires a certain penetration time (10 minutes for this set of experiments); the excess dye penetrant was removed and a developer applied. The developer helps to draw dye penetrant out of the flaw so that an indication which cannot be seen with naked eye becomes visible on the surface, to help the inspector to find the discontinuity, if exists. The key processing parameters are surface cleaning, penetration time and excess dye penetrant removing methods [31].

In this test, temperature limits are in the range 10-38°C. Also surface quality is very important to observe certain discontinuities. The first step for creating a better surface is pre-cleaning. The aim of this step is to remove all the residuals from the surface. As a surface cleaning agent, Karl Deutsch check PR-2 was used, as recommended by the ASME-Code, Section V, Article 6 standard [32]. All areas and parts were cleaned and dried before the dye penetrant's application. For that reason, the second step was drying. As the dye penetrant used in the experiment is water based, if there is any liquid residual on the surface, dye penetrant cannot disperse properly, which can cause incorrect and/or incomplete detection of cracks, pores or other discontinuities. The third step is the application of the dye penetrant. The dye penetrant ASME-Code, Section V, Article 6; Karl Deutsch RDP 1: Red dye penetrant was applied. The inspector has to be sure that all surfaces are covered with dye penetrant.

### 3.5.3. Magnetic Particle Inspection Test

Magnetic particle inspection (MPI) is a non-destructive testing method used for defect detection. It is fast and easy to use, and a specific type of surface preparation is not necessary

compared to other non-destructive tests. These characteristics make magnetic particle inspection one of the most widely utilized non-destructive testing methods.

The method is used to inspect a variety of product forms including castings, forgings and welds. Magnetic particle inspection is widely used in the structural steel construction, automotive, petrochemical, power plants, and aeronautic industries. Inspection of underwater applications such as offshore structures and underwater pipelines is another area where this method may be used.

The magnetic flux density induced in the component depends on the geometry of the component, its magnetic properties and the selection of a correct flux density is critical for the regions in which defects are expected to occur. If a flux density of the required value is established away from the prods, the value can be higher near them. The component has to be de-magnetized prior to performing magnetic particle inspections. If the component absorbs some residual magnetism, the sensitivity of the test may be lower and indications can be incorrectly obtained. The mobility of MPI particles is extremely affected by the presence of unfamiliar residues, such as dirt, rust, scale, oil or water. Some types of corrosion products will produce false indications at their boundaries [33].

In this procedure, ASTM E-1444 standard was used as reference [34]. This experiment was performed under UV light. According to the procedure, magnetic hand-yoke was supposed to be used but a magnetic coil, due to the shape of the specimen, multi directional movements and the possibility to show the defects over all the directions of the specimen, is more efficient than magnetic hand yoke. For the other non-destructive testing methods, pre-cleaning is one of the main steps. The surface of the part to be examined must be essentially smooth, clean, dry and free of residuals. The next step is applying the magnetic liquid for render visible all the defects under UV light. The magnetic inspection media was FLUXA® concentrate containing magnetic fluorescent powder. Then, the specimen was magnetized by the induced current. The procedure is accomplished by inductively coupling a part to an electrical coil to create a suitable current flow. After these steps, the inspector can see the defects and examine them under UV light. Under UV light, all the pores, defects and other discontinuities are visible in different color related to their depth. At the end, the specimen must be cleaned with certain solvents. Parts are examined to ensure that the cleaning procedure has removed magnetic particle residues from the surface defects such as crevices, holes, etc. [35].

### 3.6. Stress Crack Corrosion Test

In essence, tests for stress corrosion cracking simply require the exposure of the stressed sample of the material or component in question to the environment of interest. There are different testing methods for differing objectives:

- Standard tests (BS, ASTM, ISO and other standards for example) are generally designed to test a material for its susceptibility to SCC in a corrosive environment. For example, boiling 42%  $\text{MgCl}_2$  solution is widely used for testing the susceptibility of austenitic stainless steels to chloride stress corrosion cracking, and this test may be used to check components for the presence of residual stresses [36, 37, 38].
- Constant stress or constant displacement tests essentially describe the specimen and a loading method that stresses the specimen while exposed to the solution. The SCC susceptibility is determined by the time taken for failure of the specimen, or the crack propagation on the surface of the sample [39].
- Fracture mechanics tests use a specimen with a pre-existing crack (often made by fatigue cycling). The tests can be evaluated by recording the time to failure; however it's more common to measure the change in crack length with time, and thereby derive a graph of crack growth rate as a function of stress intensity factor,  $K_{\text{ISCC}}$  [40].

One of the important requirements for SCC is that stresses must be present in the metal. There is one method for controlling the elimination of that stresses, or at least diminishing the stresses for SCC.

The most common procedure to reduce residual stresses is annealing. With this procedure it is possible to minimize residual stresses and thus reduce the probability of cracking. This is widely applied in carbon steels, due to their high range of stresses tolerance in most environments. The same is not advisable in austenitic steels as it has a more narrow range of tolerance both in terms of temperatures and environments [41].

For large structures, this procedure is very difficult to apply; however it can be done, partially relieving stresses around welds and other risk areas.

For this test, the following standards were used:

- ASTM G36: Stress Crack Corrosion (SCC) resistance of metals and alloys in boiling  $\text{MgCl}_2$  solution [37].

- ASTM G123: Evaluating stainless steel alloys with different nickel content in boiling acidified NaCl solution [36].

Both standards were combined because of the lack of suitable equipment. The experiment procedure was based on ASTM G 36 but the test assembly was based on ASTM G123 standard. The test assembly is shown in figure 9.



Figure 9: Assembly of the SCC experiment

The aim of this test is detecting the effect of composition, heat treatment, surface finishing, microstructure and the stresses on the metal susceptibility.

Firstly, the solution has to be prepared. It contained 400 mL of distilled water and 600 grams of  $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ . Magnesium chloride must be completely dissolved in the water in order to prevent contamination. After preparing the solution, P91 specimens were placed in an Erlenmeyer flask. A condenser was used for collecting the vapor and keeping the solution in equilibrium with the solution. Next, the assembly was placed on the hot plate and the temperature was measured. The ambient conditions, regarding to the standard, have to be 1 atm and  $155 \pm 1^\circ\text{C}$ . The conditions must be constant for the reliability of the experiment.

Excessive amount of magnesium chloride must not be dumped away for safety reasons. Although  $\text{MgCl}_2$  is stable as a solid phase, safety precautions are necessary for boiling solution, which can cause severe skin burnt. For these reasons, the safety requirements must be strictly followed, and the test should be performed recurring to fume exhaustion equipment [36, 37].

## 4. Experimental Results and Discussions

In this section of the work, the results of the experimental works are presented. The experimental results are divided in three sections. These sections cover the metallographic examination, destructive, non-destructive and stress crack corrosion (SCC) tests. Tests were performed before and after welding process.

### 4.1. Metallographic analysis

Before PWHT, the examination of all the samples has proved that they present a tempered martensite structure which consists of parallel laths inside the grain.

The microstructural changes in P91 steel is based on welding process and post welding heat treatment application. Different heat treatment cycles were used to test the response of the metal in terms of structural development and hardness. Figure 10 shows the optical micrograph of the P91 steel supplied.

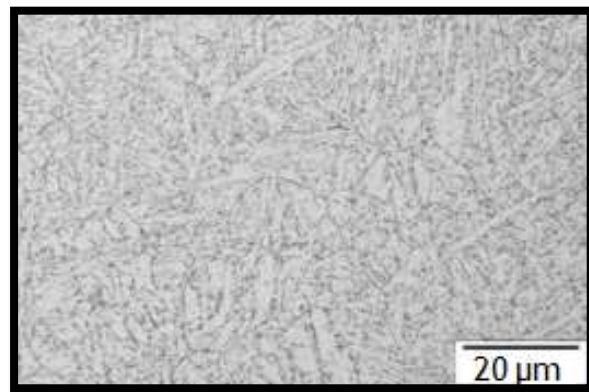


Figure 10: Microstructure of P91 steel before PWHT, etching with Vilella's reagent

During welding process, the rapid thermal cycle creates microstructural differences between base material and welding area. The amount of parameters to control welding increase the difficulty of the analysis of the effects of welding variables on weld properties. In this work, according to the induction heat treatment principles, medium frequency (6-16 kHz) was used. The heating energy is generated by an inductor. The heat is generated inside the pipe and the workpiece is heated from inside. With an excessive heat source, welding can result in unique microstructural features in different regions of the weld.

The microstructures of the samples after PWHT at different temperatures were examined. These temperatures are presented at table 6. In figure 11, microstructures of base material are

presented for different samples after PWHT at four different temperatures. In the transformation zone, specimen 1, 2, and 3 have typical fine and heterogeneous structure of P91 steel. Also, precipitates are in different size and distribution, when temperature changes. For specimen 4, temperature is out of the transformation temperature (850°C) and the microstructure changes slightly.



Figure 11: Microstructures of P91 steel base material after PWHT: 1- at 650°C; 2- at 730°C; 3- at 800°C; and 4- at 850°C, 100x, etching with Vilella's reagent

As presented in figure 12, the black spots are impurities precipitated on the grain boundaries. These precipitations cause a depletion of chromium near to the grain boundaries which makes the material anodically active. For specimen 1, the amount of carbide precipitation was lower and heterogeneously distributed. Also, the precipitates present different sizes. For specimen 2, there is a homogeneous carbide precipitation but the sizes of the precipitates are different. For sample 3, the grain growth reached the maximum value and they are dispersed on the welding zone. Finally, specimen 4, partial carbide precipitates disappeared.

Carbon and chromium contents of P91 steel increase the susceptibility of the material, however other alloying elements increase the effect of sensitization.

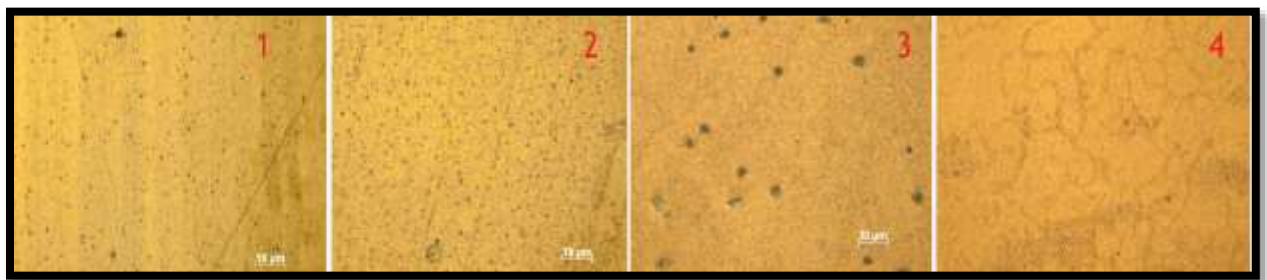


Figure 12: Microstructures of P91 steel welding zone after PWHT: 1- at 650°C; 2- at 730°C; 3- at 800°C; and 4- at 850°C, 100x, etching with Vilella's reagent



In figure 13, the microstructures of the HAZ and welding area transitions are presented. For specimen 1, carbide precipitation was disorderly distributed around the HAZ. For specimen 2, HAZ was separating the welding area and the base material clearly. In the HAZ, precipitates were homogeneous and highly concentrated. For specimen 3, it was hard to distinguish the HAZ between base material and welding area, but in the HAZ area, the size of the precipitates was fine and homogeneous. Finally, for specimen 4, HAZ area didn't present precipitates.

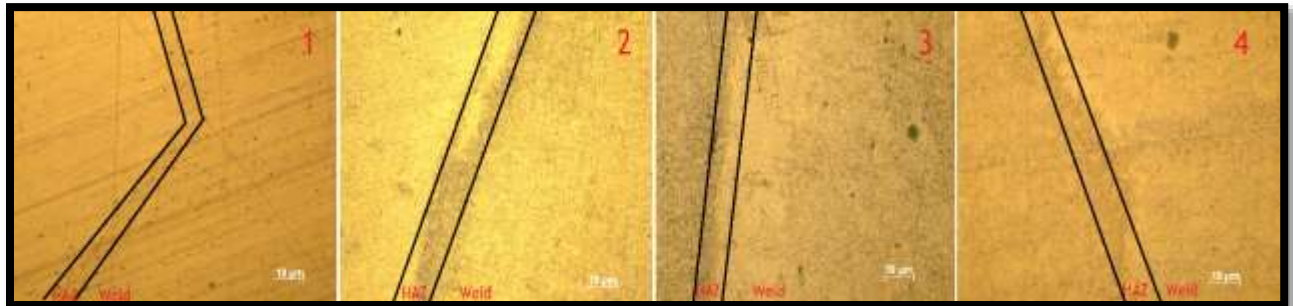


Figure 13: Microstructures of P91 steel HAZ and welding zone after PWHT: 1- at 650°C; 2- at 730°C; 3- at 800°C; and 4- at 850°C, 100x, etching with Vilella's reagent

After SCC test, first three specimen had similar structure and reaction to highly corrosive environment. Specimen 4 was heat treated above the transformation temperature and this changed the microstructure of the material and the behaviour to the SCC tests.

#### 4.2. Hardness Measurements Results

A large number of measurements have been taken to analyze the variation of the hardness across the specimens' sections. Hardness tests were performed before and after post welding heat treatment process. Hardness values are in the range from a minimum of 180 and a maximum of 300 HV, in accordance with the standard ASTM A335: High pressure boiler pipes [3].

For these measurements, 5 points are relative to the base material, heat affected zones (both side of the weld) and the welding area. These different points where the readings have been taken are shown in the figure 8.

The hardness results before PWHT were obtained with a Struers MicroHardness device. The collected hardness values from all the specimens were between the maximum and minimum limits, in accordance with the standard. Likewise, measured results are compatible with the



supplier's data presented in table 8 and appendix 6. All the measured hardness data are presented in appendixes 11, 12, 13 and 14.

Table 8: Hardness values for P91 steel from the supplier

Hardness values	Sample 1 (HV)	Sample 2 (HV)
1	235.0	232.0
2	238.0	233.0
3	236.0	235.0
Mean value	236.33	233.33

In figure 14, before and after PWHT, hardness values increased in all the areas measured. Welding zone presented the highest hardness values, as expected. The hardness measurements are between  $210\text{-}252\pm 23.3$  HV10 before PWHT, and they are slightly higher to the values  $214\text{-}283\pm 20.0$  HV10 obtained after PWHT.

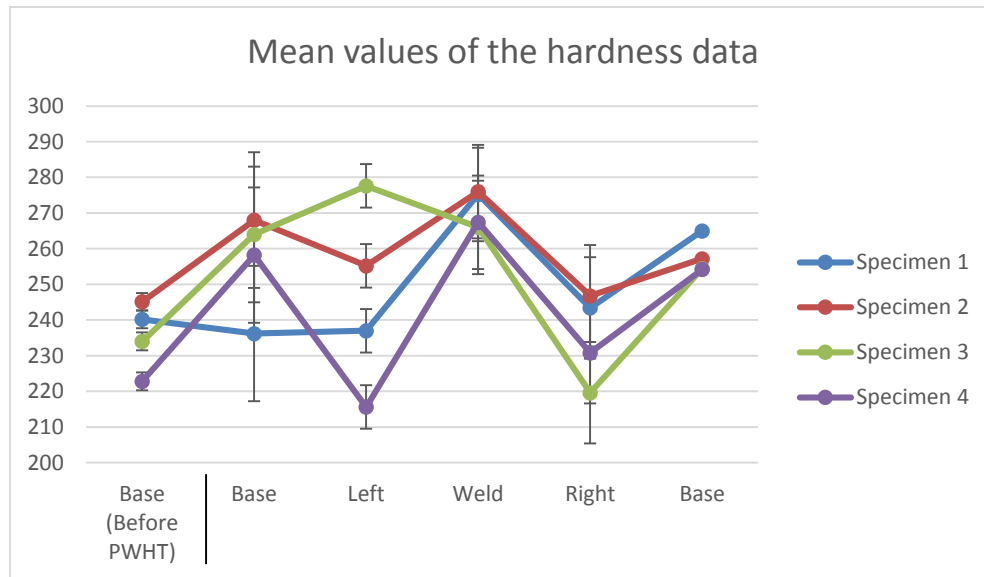


Figure 14: Hardness values of the specimens before and after PWHT

Mechanical and microstructural properties of the material are interrelated. However, the microstructural differences were not significantly reflected in the overall hardness profiles of all the samples. After PWHT, the base material and welding zone hardness values were similar; only difference was registered in the left HAZ. The difference could result from the grain size

distribution on the HAZ. For specimen 3, in left HAZ, there was finer grain size and the hardness increased.

In conclusion, all the hardness measurements before and after PWHT matched with the supplier's data as well as the standard, which means the heat treatment was successful. Also the results show that there are no significant hardness changes between the zones and they are between the maximum and minimum values of the standard.

#### 4.3. SCC test results

After finishing the sample preparation, all samples surfaces were cleaned (necessary for surface examination) and polished until a clear finishing was achieved. The dye penetrant test was performed following ASTM E 165-95: Standard test method for liquid penetrant examination standard [41].

Dye penetrant test was performed on the external and internal surfaces of the material. Firstly, as seen in Figure 15, specimens were a single piece. Then, specimens were cut; liquid penetrant test was performed again to confirm there was no crack. Dye penetrant was applied to the test material and red points around the welding area were indicating the height differences of the sample.



Figure 15: Liquid penetrant test after PWHT

As per figure 15, it was very hard to examine the inner surface of the tube. Defects were mostly located in the welded zone, but there were rare discontinuities on the base material. An attempt was made to remove these defects using abrasive discs, with most of them being removed from the surface. If the defect was deep, those specimens were separated from the others. According to the standard [31, 41], deep defects are not acceptable, because they can be a crack, thus compromising the integrity of the piece. For the SCC tests, the specimens used were those without visible surface cracks.

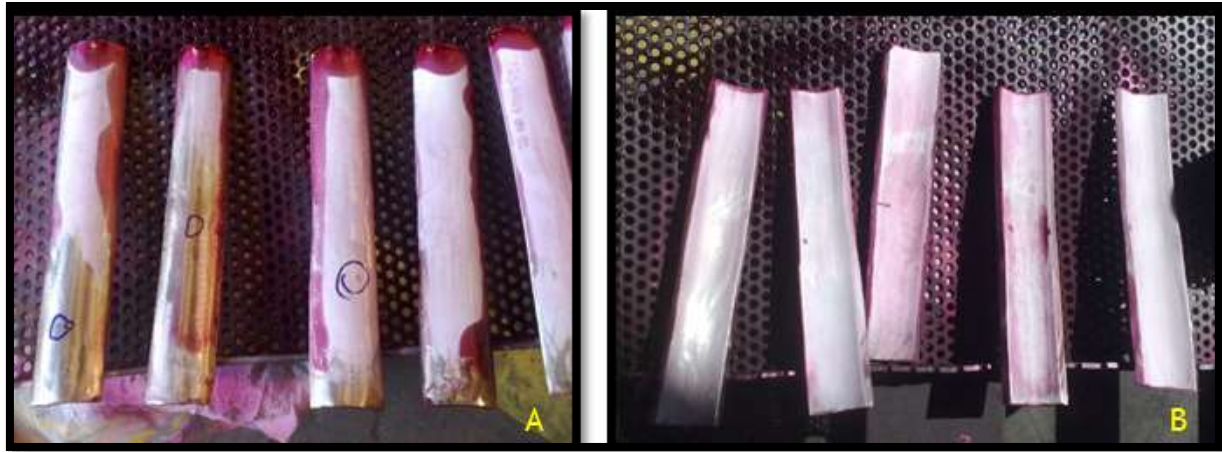


Figure 16: Liquid penetrant test after cutting the pipes: A. Outer surface; B. Inner surface

Before starting the stress crack corrosion tests, all the specimens were examined and the best ones, in terms of defect concentration, were selected. In figure 17, dark spot in specimen 1 and specimen 3 were visible, an indication of small pores as marked. They are negligible according to the standard [28, 29, 30]. Darker areas represent high intensity, (in this case, the welded areas, due to their higher intensity relative to the base material). The results of the radiation test performed before SCC are presented in appendixes 7 and 8.

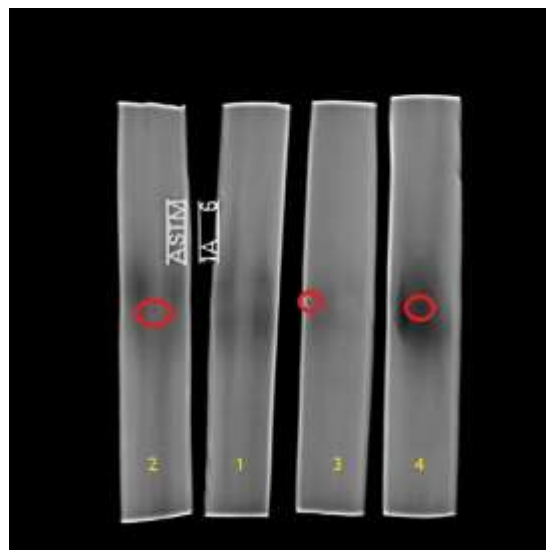


Figure 17: The results of Radiation Test of P91 steel samples, before SCC test

In the MPI test, specimen 4 had a 4.02 mm crack. According to the standard, if the crack is greater than 4.0 mm, then specimen cannot be selected. This specimen was eliminated and

replaced by another specimen. Report and the image of the MPI test before SCC test for specimen 4 are presented in appendixes 9 and 10, respectively.

At the beginning of the SCC test, all the specimens passed the magnetic particle test. Figure 18 is representing specimen 1. According to the standard [31, 33, 34], before the SCC test, defect free surface quality must be achieved. The surface was polished, and the welded area could easily distinguished.



Figure 18: MPI test of specimen 1 before SCC test

For stress crack corrosion tests, the ASTM standards G36 and G123 were used [36, 37]. The assembly belongs to the G123, and the rest of the procedure belonged to the G36 as mentioned in section 3.6. Every week, all the specimens were examined, the solution was cleaned, and all the quantitative and qualitative changes recorded.

Before SCC test, all specimens were visually examined. All specimens passed the acceptance criteria that no cracking and pitting were present. The specimens were polished to a clear surface finishing. On the surface, there was no visual discontinuity. These specimens were prepared in accordance with the guidance of ASTM G36 [37]. The length of the whole sample was 75 mm and the welding area was 12 mm long. All the specimens are presented in figure 19, before exposure to high temperature and corrosive environment.



Figure 19: Specimens before SCC tests

The weight variation test was also performed with different samples to measure the weight loss during SCC test. The solution was prepared using the same procedure for SCC test. All the samples were previously weighed and placed into the solution. During 5 weeks, all the specimens were removed from the solution, cleaned with alcohol and dried. Each sample was then weighed and the difference in weight was used to determine the corrosion rate. All the data are presented in table 9.

Table 9: Weight changes of P91 steel samples during SCC test

Sample	Initial weight (g)	Week 1 (g)	Week 2 (g)	Week 3 (g)	Week 4 (g)	Week 5 (g)	Standard Deviation
1	187.2	187.4	187.3	187.2	187.5	184.4	1.2
2	183	183.1	183.1	183	185.5	182.3	1.1
3	168.9	169.4	169.4	169.2	170.4	167.8	0.8
4	183.9	184.2	184.1	184	185.7	181.6	1.3

During the first three weeks, the experiment was evolving as expected; however on week 4, there was an incident in the laboratory and the experiment was affected; the samples were

contaminated by the  $\text{MgCl}_2$  salt and the weight increased unexpectedly. After the contamination, all the samples were cleaned with alcohol and the solution was changed for a “clean” one. After a week, the weight results were decreasing rapidly (figure 20).

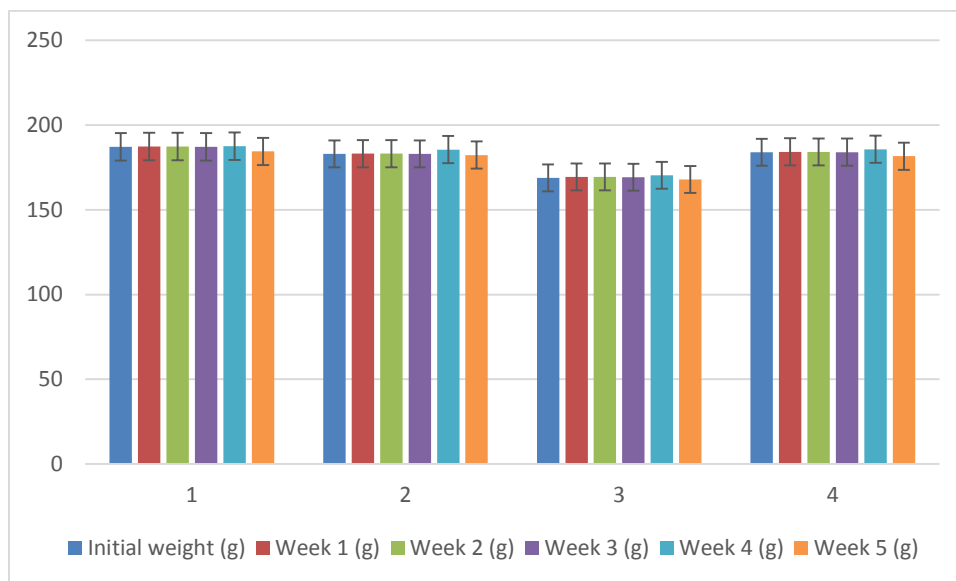


Figure 20: Weight change of P91 steel during SCC test

The specimens stayed inside the solution for 6 weeks. After this period, the visual changes on the specimens were related to the color and surface texture changes; a clearer view of the welding area was obtained (see Figure 21).



Figure 21: SCC test specimens during 6 weeks (A: week 1, B: week 2, C: week 3, D: week 4, E: week 5, F: week 6)

All specimens were inspected by using magnetic particle inspection and radiation methods after SCC test. Corrosion occurred on the surface as a thin layer. As traditional testing methods would compromise the uniformity of the material, non-destructive techniques were selected to control the corrosion on the surface.

In figure 22, sample 1 had a serious crack initiation, and if the experiment had been continued, the crack would propagate from the middle of the specimen. Sample 4 also had a point defect on the surface and the depth of the defect showed that instead of stress crack corrosion, general corrosion had occurred on the surface. Radiation test showed not only the discontinuities of the specimens after SCC tests, but also the surface defects that appeared on the surface.

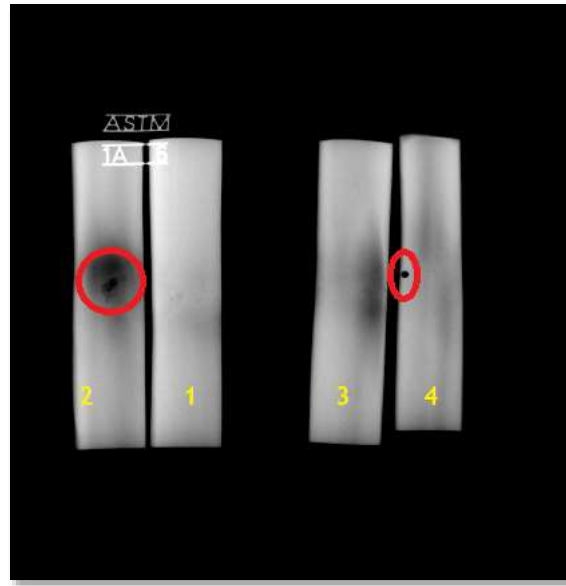


Figure 22: Radiation test results of SCC test samples after 6 weeks

After 6 weeks, all the specimens exhibited cracks. For specimen 1, the crack was detected via the radiation test. To confirm the results, MPI testing was also performed. In figure 23, the results of MPI test show that cracks occurred on the surface of the samples; the cracks were 2.52, 3.04, 2.20 and 2.86 mm long, respectively. The radiation and MPI reports after SCC testing are presented in appendixes 15 and 16.

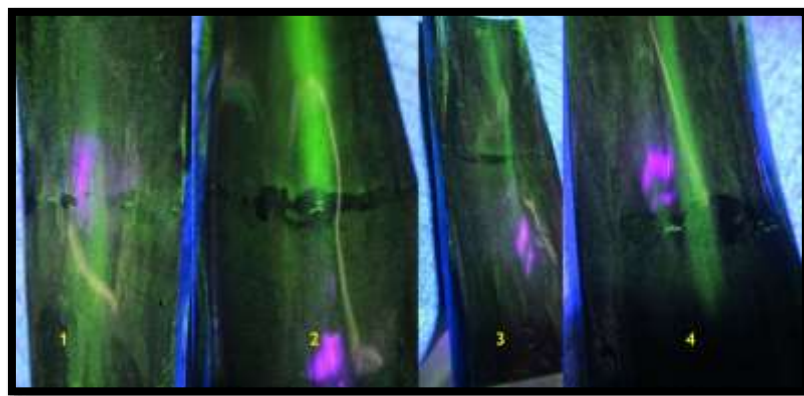


Figure 23: MPI test of specimens after SCC experiment

SCC occurring in service in welded P91 steel may be dependent of a combination of susceptibility of the weld and HAZ, and the corrosive effect of the environment. In some cases, highly aggressive environments creates general corrosion rather than SCC or neither of them.



## 5. Conclusion

After finishing the proposed study of the effect of SCC after post welding heat treatment using different temperature to welded P91 steel, the conclusions presented below were established:

- Heat treatment between 650°C and 730°C is more acceptable, because mechanical properties of P91 steel is important at high service temperature and pressure. In addition, higher hardness values in the welding zone are not desirable.
- Martensitic structure of P91 steel requires careful selection of preheat, interpass and PWHT to avoid stress crack corrosion.
- During 6 weeks of SCC experiments, weight loss and corrosion were achieved, but there are no significant findings of crack initiation. To minimize the risk of stress crack corrosion, the inspection frequency should be suitable according to the applications of the material and the capability of the facilities. This way, cracks can be detected in an early stage, and preventive methodologies applied before failure.

Some possible routes for improvement to be explored in future work may include the comparison of the effect of stress crack corrosion after PWHT with different type of welding methods such as TIG versus SMAW.

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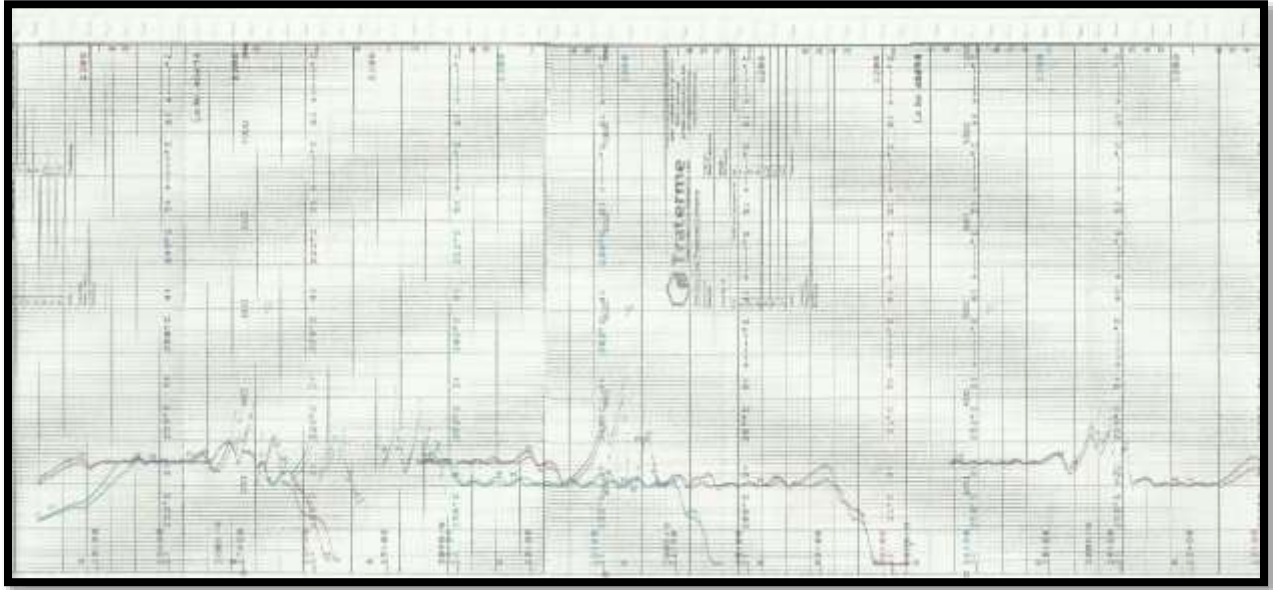
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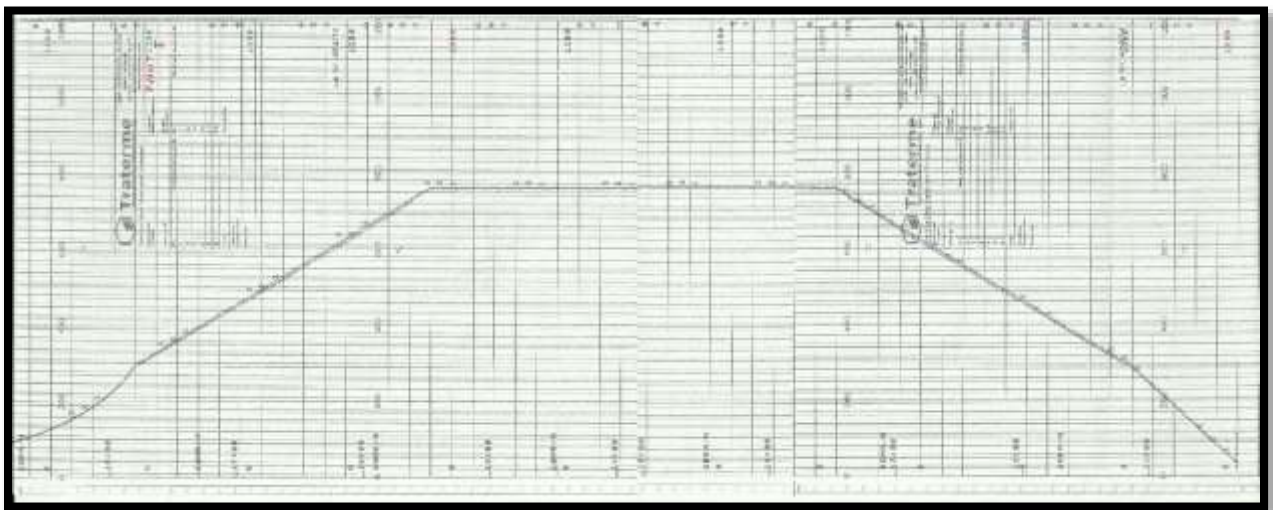
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## Appendixes

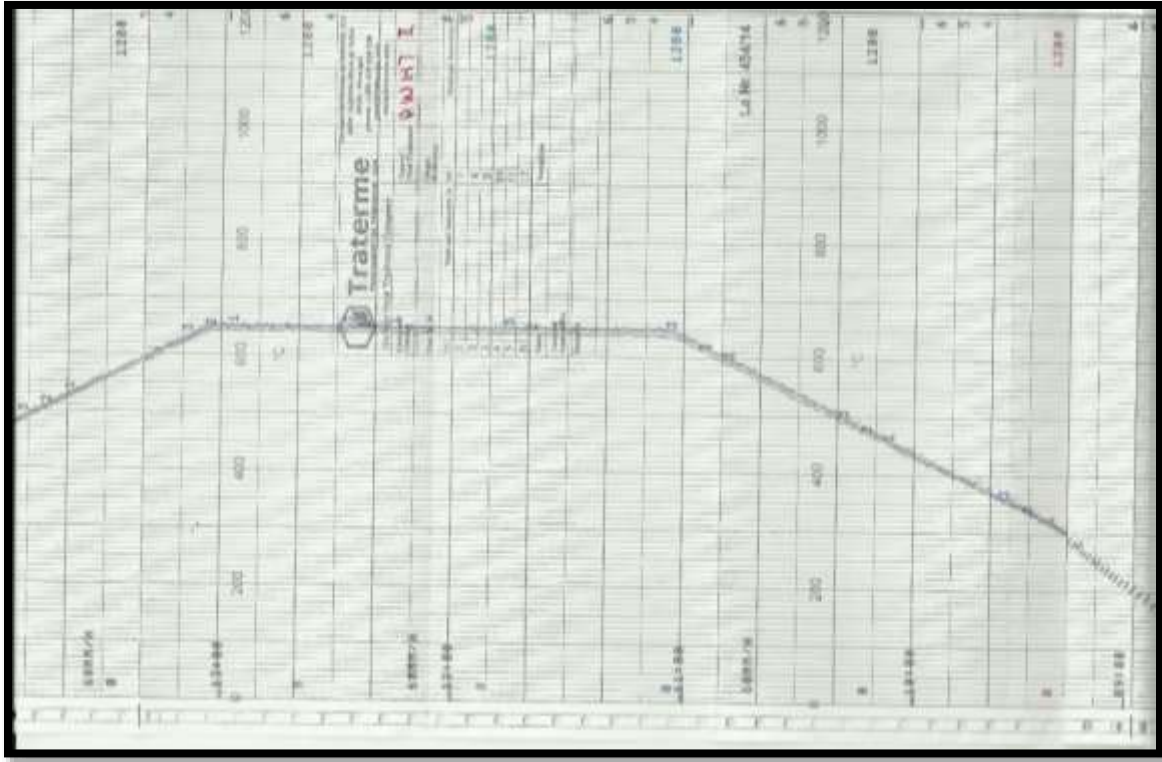
### 1. Graph of the Preheating and Interpass Heating



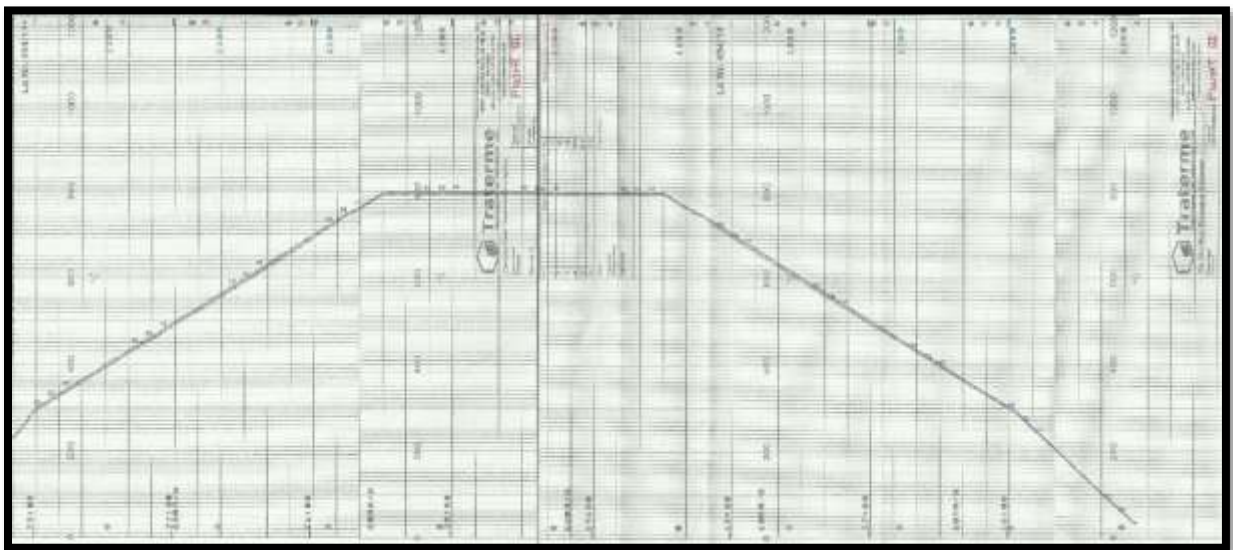
### 2. Graph of the PWHT of Specimen 1



3. Graph of the PWHT of Specimen 2

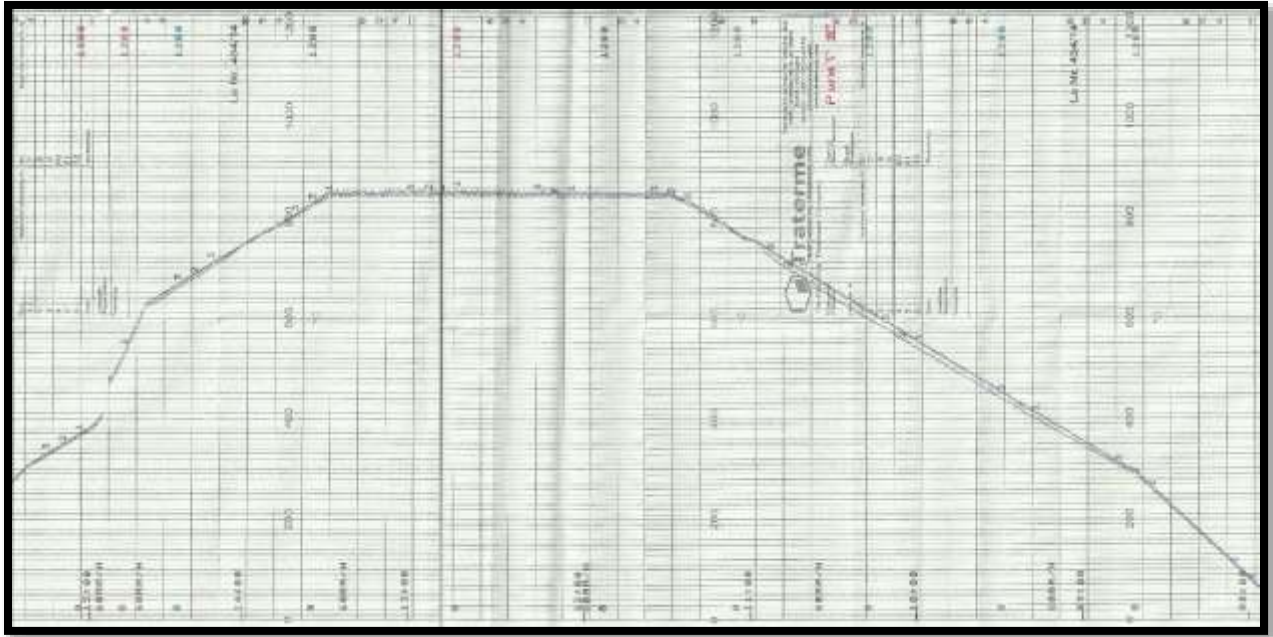


4. Graph of the PWHT of Specimen 3





5. Graph of the PWHT of Specimen 4



Selin ÇOKGÜL

6204-050-0712R01300\_P15\_M45\_CH412810



# INSPECTION CERTIFICATE

ABNAHMEPRÜFZEUGNIS

(UNI EN 10204 3.1 / ISO 10474 3.1.B)

Number / Nummer: 02/12/20346

Page / Seite: 2 / 5

Date / Datum: September 20, 2012

Sheet 5 of 10  
13. März 2012  
480117 Zerk. (24)  
Rev. 0  
Tel: +49 203 56739  
Fax: +49 203 56734

Customer / Kunde: FERROSTAAL / PVRING SUPPLY GMBH

Manufacturing Process / Herstellungsprozess: NORMALIZED AND TEMPERED

NORMALIZED AND TEMPERED

Standard or Specification / Norm oder Spezifikation: Standard of Specification / Norm oder Spezifikation: ACC. TO ASME SA 335, ASTM A 335 + TCC 20019608 REV. 2.2.

GER. ASME SA 335, ASTM A 335 + TCC 20019608 REV. 2.2.

Dimensions / Abmessungen: 1.300" O.D. x 200" W.T.

Product Type / Art des Produkts: SEAMLESS HOT FINISHED STEEL TUBES FOR BOILER (WITH EXTRA REQUIREMENTS)

SEAMLESS HOT FINISHED STEEL TUBES FOR BOILER (WITH EXTRA REQUIREMENTS)

Product Type / Art des Produkts: NAHTLOSE QUALITÄTSTAHL-RÖHRE WASSERFÜHRT ALS KESSELROHRE KEIN STÄNDIG

Product Type / Art des Produkts: STEEL, OR P91, ASTM A 335/ASME SA 335

STEEL, OR P91, ASTM A 335/ASME SA 335

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## CHEMICAL COMPOSITION / CHEMISCHE ZUSAMMENSETZUNG

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		a wt%							b wt%						
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673203	Sample No.	H	Min	0.2	0.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Product No.	H	Max	0.3	0.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
673203	Sample No.	H	Min	0.2	0.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Product No.	H	Max	0.3	0.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
673203	Sample No.	H	Min	0.2	0.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Product No.	H	Max	0.3	0.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
673203	Sample No.	H	Min	0.2	0.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Product No.	H	Max	0.3	0.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
673203	Sample No.	H	Min	0.2	0.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Product No.	H	Max	0.3	0.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
673203	Sample No.	H	Min	0.2	0.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Product No.	H	Max	0.3	0.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
673203	Sample No.	H	Min	0.2	0.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Product No.	H	Max	0.3	0.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
673203	Sample No.	H	Min	0.2	0.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Product No.	H	Max	0.3	0.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
673203	Sample No.	H	Min	0.2	0.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Product No.	H	Max	0.3	0.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
673203	Sample No.	H	Min	0.2	0.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Product No.	H	Max	0.3	0.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
673203	Sample No.	H	Min	0.2	0.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Product No.	H	Max	0.3	0.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
673203	Sample No.	H	Min	0.2	0.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Product No.	H	Max	0.3	0.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
673203	Sample No.	H	Min	0.2	0.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Product No.	H	Max	0.3	0.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
673203	Sample No.	H	Min	0.2	0.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Product No.	H	Max	0.3	0.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
673203	Sample No.	H	Min	0.2	0.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Product No.	H	Max	0.3	0.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
673203	Sample No.	H	Min	0.2	0.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Product No.	H	Max	0.3	0.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
673203	Sample No.	H	Min	0.2	0.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Product No.	H	Max	0.3	0.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
673203	Sample No.	H	Min	0.2	0.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Product No.	H	Max	0.3	0.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
673203	Sample No.	H	Min	0.2	0.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Product No.	H	Max	0.3	0.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
673203	Sample No.	H	Min	0.2	0.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Product No.	H	Max	0.3	0.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
673203	Sample No.	H	Min	0.2	0.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Product No.	H	Max	0.3	0.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
673203	Sample No.	H	Min	0.2	0.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Product No.	H	Max	0.3	0.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
673203	Sample No.	H	Min	0.2	0.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Product No.	H	Max	0.3	0.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
673203	Sample No.	H	Min	0.2	0.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Product No.	H	Max	0.3	0.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
673203	Sample No.	H	Min	0.2	0.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Product No.	H	Max	0.3	0.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
673203	Sample No.	H	Min	0.2	0.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Product No.	H	Max	0.3	0.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
673203	Sample No.	H	Min	0.2	0.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Product No.	H	Max	0.3	0.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
673203	Sample No.	H	Min	0.2	0.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Product No.	H	Max	0.3	0.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
673203	Sample No.	H	Min	0.2	0.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Product No.	H	Max	0.3	0.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
673203	Sample No.	H	Min	0.2	0.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Product No.	H	Max	0.3	0.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
673203	Sample No.	H	Min	0.2	0.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Product No.	H	Max	0.3	0.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
673203	Sample No.	H	Min	0.2	0.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Product No.	H	Max	0.3	0.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
673203	Sample No.	H	Min	0.2	0.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Product No.	H	Max	0.3	0.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
673203	Sample No.	H	Min	0.2	0.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Product No.	H	Max	0.3	0.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
673203	Sample No.	H	Min	0.2	0.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Product No.	H	Max	0.3	0.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
673203	Sample No.	H	Min	0.2	0.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Product No.	H	Max	0.3	0.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
673203	Sample No.	H	Min	0.2	0.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Product No.	H	Max	0.3	0.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
673203	Sample No.	H	Min	0.2	0.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Product No.	H	Max	0.3	0.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0	



# INSPECTION CERTIFICATE ABNAHMEPRUEFZEUGNIS (UNI EN 10204 3.1 / ISO 10474 3.1B)

Number / Nummer: 0212/20346  
Page / Seite: 3 / 5  
Date / Datum: September 20, 2012

Seiten 3/5  
Muster-Nr. 0212/20346  
Rev. 04  
Tel. +49 240 83723  
Fax. +49 240 161161

Customer / Kunde: FERROVIAL PIPING SUPPLY GmbH

Product Type / Art des Produkts: SEAMLESS HOT FINISHED STEEL TUBES FOR BOILER (WITH EXTRA REQUIREMENTS)

Customer's Material / Kunden Material-Nr.: 45001871402050271300

Manufacturer's Order / Auftrags-Nr.: 2263001005

Normalizing Process / Normalisierungsvorgang: NORMALIZED AND TEMPERED

Seamless Hot Finished Steel Tubes for Boiler (with extra requirements)

Surface / Oberfläche: INT AND EXT BARE

Standard / Norm oder Spezifikation: ASME SA 335, ASTM A 335 + T0C 20019008 REV. 2.2.

Seamless Hot Finished Steel Tubes for Boiler (with extra requirements)

Ends / Enden: PLAIN ENDS (SQUARE CUT)

Dimensions / Abmessungen: 11320.7 ID, 11320.7 OD, 5.41 WALL THK, 11320.7 ID, 11320.7 OD, 5.41 WALL THK

Seamless Hot Finished Steel Tubes for Boiler (with extra requirements)

Plain Ends (Square Cut)

11320.7 O.D. x 5.41 WALL THK

Length / Länge: 11100 mm Flared LG

Weight / Gewicht: 5135 kg

Weight / Gewicht: 5.41 Kg/m

## = IMPACT TEST / KERBSCHLAGPROBE =

Position / Position	Temp. / Temp.	Charpy V	Charpy V	Charpy V
Test Nr.	Zone	LA	3c	Zone
Charpy Nr.	Probe Nr.	1	2	3
412318	BS3018	27.0	28.0	27.0

Any Average / Mittelwert: 27.0  
Body / Körper: L1 longitudinal / L2 longitudinal  
Test Method / Testverfahren: Charpy V

## = FLATTENING TEST / FALTVERSUCH =

Standard / Standard						
Test Nr.	Sample Nr.	Zone	Sc	Test Frequency	Result	
Charpy Nr.	Probe Nr.	Zone		Frequency	Epilotic	
412318	BS3018	1	2	3		

Standard / Standard						
Test Nr.	Sample Nr.	Zone	Sc	Test Frequency	Result	
Charpy Nr.	Probe Nr.	Zone		Frequency	Epilotic	
412318	BS3018	1	2	3		

Test Method / Testverfahren: Charpy V

Any Average / Mittelwert: 27.0  
Body / Körper: L1 longitudinal / L2 longitudinal  
Test Method / Testverfahren: Charpy V



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**INSPECTION CERTIFICATE**  
**ABNAHMEPRUEFZEUGNIS**  
(UNI EN 10204 3.1 / ISO 10474 3.1.B)

Number / Nummer: **02/12/20346**  
Page / Seite: **4 / 5**  
Date / Datum: **September 28, 2013**

Steelco S.A. Prod.  
St. Joris-Windisch  
42011 Ebnat, Switzerland  
Tel.: +41 20 801 80 00  
Fax: +41 20 801 80 01

Customer / Kunde: <b>HERNOSTAL PIPING SUPPLY GMBH</b>	Product Type / Artikelbezeichnung: <b>SEAMLESS HOT FINISHED STEEL TUBES FOR BOILER (WITH EXTRA REQUIREMENTS)</b>	Customer's Reference / Kunden-Referenz: <b>3263081005</b>	Manufacturer's Marking / Hersteller-Markierung: <b>INT AND EXT BARE PROSCHWARZ</b>
Normalizing Process / Normalisierungsprozess: <b>NORMALIZED AND TEMPERED</b>	Standard / Norm: <b>ASME SA 333, ASTM A 333 + TOC 20019008 REV. 2.2.</b>	Standard / Norm: <b>ASME SA 333, ASTM A 333 + TOC 20019008 REV. 2.2.</b>	Standard / Norm: <b>ASME SA 333, ASTM A 333 + TOC 20019008 REV. 2.2.</b>
Dimensions / Abmessungen: <b>Ø 1.027" O.D. x .201" W.T.</b>	Length / Länge: <b>11100 mm Fixed Lg</b>	Quantity / Menge: <b>11320.7 lb</b>	Weight / Gewicht: <b>5.41 kg/m</b>
Dimensions / Abmessungen: <b>Ø 48.3mm O.D. x .510mm W.T.</b>	Length / Länge: <b>11100 mm Fixed Lg</b>	Quantity / Menge: <b>8135 kg</b>	Weight / Gewicht: <b>5.41 kg/m</b>

**SUPPLEMENTARY INFORMATION / ZUSÄTZLICHE INFORMATIONEN**

**STANDARD ENTRIES**  
**AUSGABE EINTRÄGE**

Customer / Kunde: <b>Steelco S.A.</b>	Product Type / Artikelbezeichnung: <b>SEAMLESS HOT FINISHED STEEL TUBES FOR BOILER (WITH EXTRA REQUIREMENTS)</b>	Customer's Reference / Kunden-Referenz: <b>3263081005</b>	Manufacturer's Marking / Hersteller-Markierung: <b>INT AND EXT BARE PROSCHWARZ</b>
Normalizing Process / Normalisierungsprozess: <b>NORMALIZED AND TEMPERED</b>	Standard / Norm: <b>ASME SA 333, ASTM A 333 + TOC 20019008 REV. 2.2.</b>	Standard / Norm: <b>ASME SA 333, ASTM A 333 + TOC 20019008 REV. 2.2.</b>	Standard / Norm: <b>ASME SA 333, ASTM A 333 + TOC 20019008 REV. 2.2.</b>

**Supplementary Information**  
**Zusätzliche Informationen**

NOTES: 1. PRODUCT TYPE: SEAMLESS HOT FINISHED STEEL TUBES FOR BOILER (WITH EXTRA REQUIREMENTS). 2. STANDARD: ASME SA 333, ASTM A 333 + TOC 20019008 REV. 2.2. 3. DIMENSIONS: Ø 1.027" O.D. x .201" W.T. 4. LENGTH: 11100 mm Fixed Lg. 5. QUANTITY: 11320.7 lb. 6. WEIGHT: 5.41 kg/m. 7. MANUFACTURER'S MARKING: INT AND EXT BARE PROSCHWARZ. 8. NORMALIZING PROCESS: NORMALIZED AND TEMPERED. 9. STANDARD ENTRIES: SEE SUPPLEMENTARY INFORMATION. 10. OTHER INFORMATION: SEE SUPPLEMENTARY INFORMATION.	NOTES: 1. PRODUCT TYPE: SEAMLESS HOT FINISHED STEEL TUBES FOR BOILER (WITH EXTRA REQUIREMENTS). 2. STANDARD: ASME SA 333, ASTM A 333 + TOC 20019008 REV. 2.2. 3. DIMENSIONS: Ø 1.027" O.D. x .201" W.T. 4. LENGTH: 11100 mm Fixed Lg. 5. QUANTITY: 11320.7 lb. 6. WEIGHT: 5.41 kg/m. 7. MANUFACTURER'S MARKING: INT AND EXT BARE PROSCHWARZ. 8. NORMALIZING PROCESS: NORMALIZED AND TEMPERED. 9. STANDARD ENTRIES: SEE SUPPLEMENTARY INFORMATION. 10. OTHER INFORMATION: SEE SUPPLEMENTARY INFORMATION.
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This certificate is issued by a company whose products are used in the construction of pressure vessels. The user of this certificate is responsible for the correct use of the certificate. The user of this certificate is responsible for the correct use of the certificate. The user of this certificate is responsible for the correct use of the certificate.



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(UNI EN 10204 3.1 / ISO 10474 3.1.B)

Number / Nummer:	Page / Seite:
02/12/20346	5 / 5
Date / Datum:	
September 20, 2012	

**Director, G.N. Prasad**  
 03, Ashok Vihar, New Delhi  
 110021 India, 110041

**Phone:**  
 Tel. + 91 260 520720  
 Fax + 91 260 661511

DISCUSSION / Possible PERIODONTAL TISSUE SUPPLY GROWN

450018714072055271300

Chenier's *Notion of Murder: Relativity*

2263054/005

www.fishbase.org

Product Type / Art. Code / Product Name

NORMALIZED AND TEMPERED  
NORMALGEGLUT UND ANLASSEN

SEAMLESS HOT FINISHED STEEL TUBES FOR BOILER (WITH EXTRA REQUIREMENTS)  
NAHTLOSBE QUALITÄTSTAHLROHRE WÄRMEGEFERTIGT ALS KESSELROHRE (KEIN STANDARD)

Continued on opposite page

Name: \_\_\_\_\_

ACC. TO ASME SA 335, ASTM A 335 + TQC 2001B608 REV. 2.2.

STEEL 0

GEN. ASME & A 33, ASTM A 33

1

Discussions / Addressing:

$$\text{Length}(\text{in}) / \text{Length}(\text{in}) =$$

0.413mm O.D. X 5.08mm W.T.

11100 mm Fixed leg

MARKING / MARKIERUNG

Admission

LACKERUNG

**Abstract**

Capacitance intervals 5–50 pF or elem; CAPUT 1; TSI; ASTIMUSWE AVSA 330; P91

HENRI JACQUES LAFONT  
+39018714036552711300

127

#### LEGEND

This is to certify that no product described here has been manufactured, sampled, tested and inspected in accordance with the applicable product requirements. This certificate is not a declaration of origin nor may it be used as a declaration of origin.

Hierzu wird festgelegt, dass das im Absatz beschriebene Merkmal gemessen der Anforderung an die Bestimmung zugeordnet ist. Dieses Absatz ist kein Umformungsanforderung und kann nicht als solche verwendet werden.

CUSTOMER, THIRD PARTY

TENARIS QUALITY DEPARTMENT SIGNATURE

INSPECTION COMPANY  
ADVISORIAL BOARD

*L. sordida*

QUANTITATIVE CERTIFICATION DEPT.  
FACTS-AND-FIGURES-ADDITIONAL

OLAVI V. MEIKERILÄ, ANNE KALLI  
VERANTWORTLICHER QUALITÄTSMANAGER  
MOJOINT™ Järvisalo

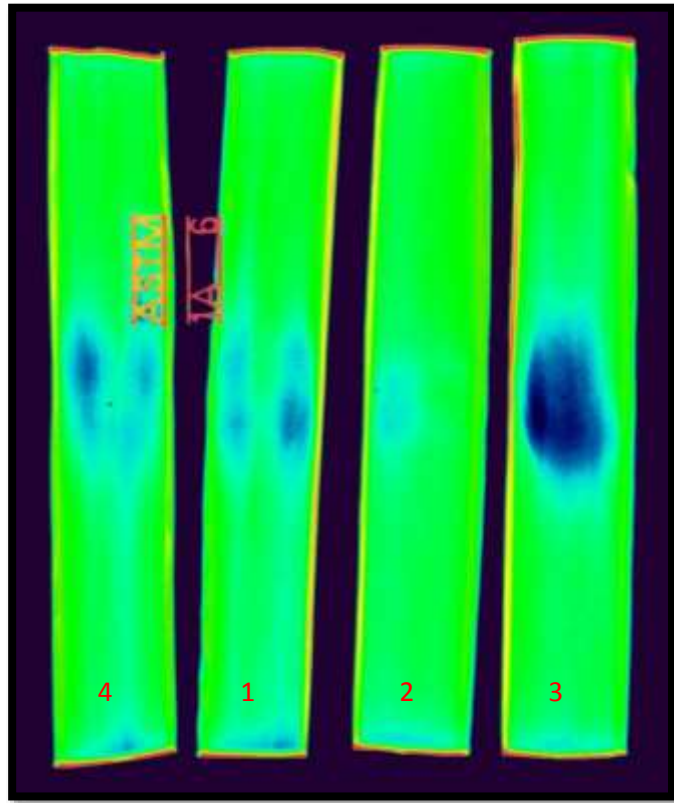
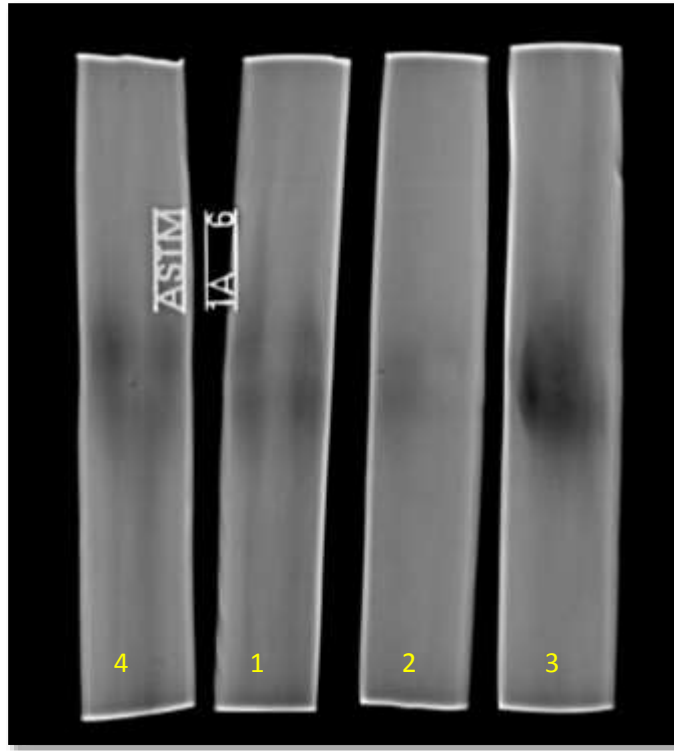
The contents of this e-mail are confidential for you and it is valid only for the person signatory. On the original signature, the text must give evidence of the signature. In case the content of the signed electronic message reflects a copy of it, the result shall be considered as having been received by the responsible for the original or not signed one. Any absolute and/or limitation will be subjected to the law.

[illegible]

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7. Picture of the Radiation Test Samples before SCC test







Selin ÇOKGÜL

X



9. Magnetic Particle Analysis Report before SCC test

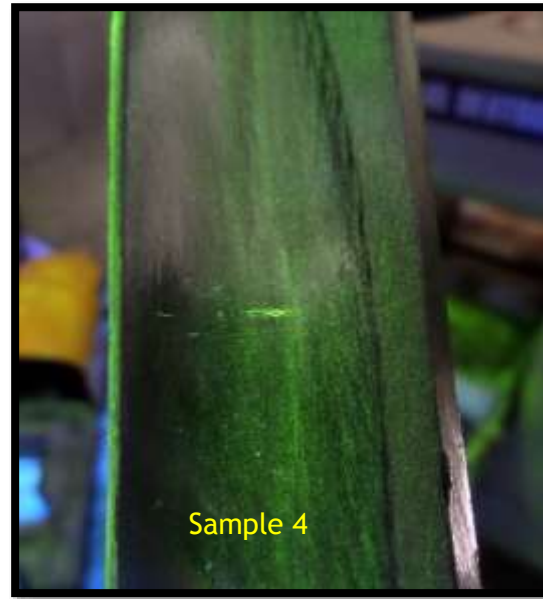
 <b>RELATÓRIO DE ENSAIO POR PARTÍCULAS MAGNÉTICAS</b> <b>MAGNETIC PARTICLE TESTING REPORT</b>			
<b>Cliente:</b> <b>Client:</b> <b>Local:</b> <b>Pólo:</b>	<b>Tratame, Lda - Travessa do Campo da</b> <b>Telhira, 211 - Vila Nova da Telha</b> <b>A. Jorge Lima, Lda.</b> <b>Rua da Serralves, 328 - Porto</b>	<b>Encomenda nº:</b> <b>Order no:</b> <b>Nº Relatório:</b> <b>Report no:</b>	<b>OS 272 (2014)</b>  <b>MPY-14-110</b>
<b>Identificação peça(s):</b> <b>Part(s) identification:</b> Sample 1 to 4			
<b>Tipo de componente:</b> <b>Component type</b> <input type="checkbox"/> Material Base <b>Base Material</b> <input checked="" type="checkbox"/> Soldado <b>Welded</b> <input type="checkbox"/> Forjado <b>Forged</b> <input type="checkbox"/> Fundido <b>Casted</b>			
<b>Fase da inspeção:</b> <b>Inspection moment</b> <input type="checkbox"/> Em produção <b>In production</b> <input type="checkbox"/> Em serviço <b>In service</b> <input type="checkbox"/> Antes Trat. Térmico <b>Before Heat Treat.</b> <input checked="" type="checkbox"/> Depois Trat. Térmico <b>After Heat Treat.</b>			
<b>Área controlada:</b> <b>Inspected area:</b> 100%		<b>Desenhos aplicáveis:</b> <b>Drawing no:</b> —	
<b>Material:</b> <b>Material:</b> P91		<b>Iluminância (lux):</b> <b>Illuminance (lux):</b> < 20	
<b>Preparação superfície:</b> <b>Surface condition:</b> Desengordurada <b>Depressed</b>		<b>Irradiância (µW/cm²):</b> <b>Irradiance (µW/cm²):</b> > 1000	
<b>Técnica de magnetização:</b> <b>Magnetizing Technique:</b> Bobine		<b>Método do ensaio:</b> <b>Test method:</b> Contínuo <b>Continuous</b>	
<b>Equipamento:</b> <b>Equipment:</b> MTQ16		<b>Corrente (AC/DC):</b> <b>Current (AC/DC):</b> AC	
<b>Espaçamento entre pontas:</b> <b>Not spacing</b> —		<b>Intensidade da corrente:</b> <b>Current intensity:</b> —	
<b>Força do campo tangencial:</b> <b>Tangential field strength:</b> > 2,4 kA/m		<b>Indicador de campo:</b> <b>Field indicator:</b> Berthold	
<b>Tipo de partículas:</b> <b>Type of particles:</b> Fluorescentes/Via húmida <b>Fluorescents/Wet</b>		<b>Tinta de contraste:</b> <b>Contrast paint:</b> Não <b>No</b>	
<b>Referência:</b> <b>M. Particles Reference:</b> KD Flux Suspension HS-O <b>Lot/Batch:</b> 2191		<b>Desmagnetização:</b> <b>Demagnetization:</b> Não <b>No</b>	
<b>Normas de ensaio:</b> <b>Testing standards:</b> ASME BPVC-V-2010+A1:2011 [ART. 7]			
<b>Critério de aceitação:</b> <b>Evaluation references:</b>		<b>Normas:</b> <b>Standards:</b> N/A	<b>Classe:</b> <b>Class:</b> N/A
<b>Resultados</b> <b>Results</b>		<b>Observações</b> <b>Remarks</b>	
<b>Aceitável</b> <b>Acceptable</b> <input type="checkbox"/>		<b>Magnetização feita em múltiplas direcções.</b> <b>Magnetization done in multiple directions.</b>  <b>Sample nº 4 had 4,0 mm length cracks</b>	
<b>Não aceitável</b> <b>Not acceptable</b> <input type="checkbox"/>			
<b>Aceitável com registos</b> <b>Acceptable with notes</b> <input checked="" type="checkbox"/>			
<b>OPERADOR</b> <b>TECHNICIAN</b>  <b>NOME/NAME:</b> António Lima <b>Nº CERT./CERT. NO:</b> END 5620 <b>CARGO/POSITION:</b> Level II <b>RUBRICA E CARIMBO/STAMP:</b>		<b>RESPONSÁVEL</b> <b>RESPONSIBLE</b>  <b>NOME/NAME:</b> António Lima <b>Nº CERT./CERT. NO:</b> END 373 <b>CARGO/POSITION:</b> Level III <b>RUBRICA E CARIMBO/STAMP:</b>	
		<b>DATAS</b> <b>DATES</b> <b>INÍCIO/BEGINNING:</b> 23 de Maio de 2014 <b>FIM/CLOSURE:</b> 23 de Maio de 2014 <b>VERIFICAÇÃO/VERIFICATION:</b> 26 de Maio de 2014	

Mod. PQ10.03/07

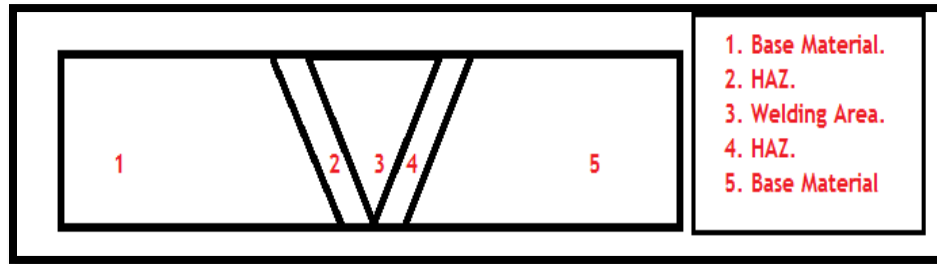
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 Tel: (+351) 226 155 257 / Fax: (+351) 226 181 098 / email: a.jorge.lima@ipac.pt / www.a.jorge.lima.com

Página/Page: 1 de 1

#### 10. MPI Test Samples before SCC



#### 11. Hardness Values of the Specimen 1, before and after PWHT



This drawing represents the indentation zones of hardness measurements. In each zones, 5 hardness values were registered.

Hardness Tests before PWHT					
Specimen 1					
	1	2	3	4	5
Random	232.5	239.3	239.3	239.3	239.3
Random	222.2	228.5	243.6	261.8	255.5
Average	227.4	234	241.5	250.6	247.4
Standard Deviation	7.28	7.64	3.04	15.91	11.46
Hardness Tests After PWHT					

Specimen 1					
	1	2	3	4	5
Base	232	238	237	238	236
Left	255	214	257	235	224
Weld	273	268	278	284	273
Right	257	232	245	255	228
Base	263	250	287	256	269
Average	236.6	237.4	243.4	243.4	265
Standard Deviation	2.43	18.75	6.20	13.12	14.23

## 12. Hardness Values of Specimen 2, before and after PWHT

Hardness Tests with Before PWHT					
	1	2	3	4	5
Random	220.9	223.3	218.4	224.6	220.9
Random	268.1	270	258.6	258.6	287.4
Average	244.5	246.65	238.5	241.6	254.15
Standard Deviation	33.38	33.02	28.43	24.04	47.02
Hardness Tests with After PWHT					
	1	2	3	4	5
Base	289	262	251	239	299
Left	273	282	235	228	258
Weld	285	300	235	265	298
Right	229	228	242	262	273
Base	297	229	279	210	271
Average	268	255.2	276.6	246.8	257.2
Standard Deviation	25.33	23.40	27.11	20.1	36.3

## 13. Hardness Values of Specimen 3, before and after heat treatment

Hardness Tests with Before PWHT					
	1	2	3	4	5
Random	213.6	211.3	212.5	205.6	209

Random	252.4	268.2	266.5	245	255.4
Average	233	239.75	239.5	225.3	232.2
Standard Deviation	27.44	40.23	38.18	27.86	32.81
<b>Hardness Tests with After PWHT</b>					
	1	2	3	4	5
Base	275	282	288	228	247
Left	286	288	271	294	249
Weld	276	292	286	218	258
Right	218	228	242	204	206
Base	258	247	241	255	271
Average	262.6	267.4	265.6	239.8	246.2
Standard Deviation	26.8	28.3	22.96	35.6	24.4

14. Hardness Values of Specimen 4, before and after heat treatment

<b>Hardness Tests with Before PWHT</b>					
	1	2	3	4	5
Random	213.6	211.3	212.5	205.6	209
Random	227.3	230	235.2	235.2	248
Average	220.45	220.65	223.85	220.4	228.5
Standard Deviation	9.69	13.22	16.05	20.93	27.58
<b>Hardness Tests with After PWHT</b>					
	1	2	3	4	5
Base	258	287	239	224	283
Left	225	217	221	204	211
Weld	270	307	256	244	260
Right	229	225	223	221	256
Base	214	284	261	297	215
Average	239.2	264	240	238	245
Standard Deviation	23.7	40.3	18.4	35.9	31

15. Magnetic Particle Analysis Report after SCC test



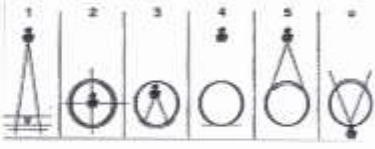


 <b>RELATÓRIO DE ENSAIO POR PARTÍCULAS MAGNÉTICAS</b> MAGNETIC PARTICLE TESTING REPORT			
<b>Cliente:</b> <b>Client:</b> <b>Local:</b> <b>Place:</b>	<b>Traterme, Lda - Travessa do Campo da Telheira, 211 – Vila Nova da Telha</b> <b>A.Jorge Lima, Lda.</b> Rua de Serralves, 328 - Porto	<b>Encomenda nº:</b> <b>Order no:</b>	<b>OS 272 (2014)</b>
		<b>Nº Relatório:</b> <b>Report no:</b>	<b>MPT-14-125</b>
<b>Identificação peça(s):</b> <i>Part(s) identification:</i>			
Sample 1 to 4			
<b>Tipo de componente:</b> <i>Component type:</i>			
<input type="checkbox"/> Material Base Base Material			
<input checked="" type="checkbox"/> Soldado Welded			
<input type="checkbox"/> Forjado Forged			
<input type="checkbox"/> Fundido Casted			
<b>Fase da inspeção:</b> <i>Inspection moment:</i>			
<input type="checkbox"/> Em produção In production			
<input type="checkbox"/> Em serviço In service			
<input type="checkbox"/> Antes Trat. Térmico Before Heat Treat.			
<input checked="" type="checkbox"/> Depois Trat. Térmico After Heat Treat.			
<b>Área controlada:</b> <i>Inspected area:</i>		<b>Desenhos aplicáveis:</b> <i>Drawing no:</i>	
100%		—	
<b>Material:</b> <i>Material:</i>		<b>Iluminância (lux):</b> <i>Illuminance (lux):</i>	
P91		< 20	
<b>Preparação superfície:</b> <i>Surface condition:</i>		<b>Irradiância (µW/cm²):</b> <i>Irradiance (µW/cm²):</i>	
Desengordurada Degreased		> 1000	
<b>Técnica de magnetização:</b> <i>Magnetizing Technique:</i>		<b>Método do ensaio:</b> <i>Test method:</i>	
Bobine		Contínuo Continuous	
<b>Equipamento:</b> <i>Equipment:</i>		<b>Corrente (AC/DC):</b> <i>Current (AC/DC):</i>	
MT016		AC	
<b>Espaçamento entre pontas:</b> <i>Rod spacing:</i>		<b>Intensidade da corrente:</b> <i>Current intensity:</i>	
—		—	
<b>Força do campo tangencial:</b> <i>Tangential field strength:</i>		<b>Indicador de campo:</b> <i>Field indicator:</i>	
> 2,4 kA/m		Berthold	
<b>Tipo de partículas:</b> <i>Type of particles:</i>		<b>Tinta de contraste:</b> <i>Contrast paint:</i>	
Fluorescentes/Via húmida Fluorescents/Wet		Não No	
<b>Referência:</b> <i>M. Particles Reference:</i>		<b>Desmagnetização:</b> <i>Desmagnetizing:</i>	
KD Fluxa Suspension HS-O Lots/Batch: 2191		Não No	
<b>Normas de ensaio:</b> <i>Testing standards:</i>			
ASME BPVC-V-2010+A1:2011 (ART. 7)			
<b>Critério de aceitação:</b> <i>Evaluation references:</i>		<b>Norma:</b> Standard:	<b>Classe:</b> Class:
N/A		N/A	
<b>Resultados</b> <i>Results</i>		<b>Observações</b> <i>Remarks</i>	
<b>Aceitável</b> Acceptable		<input type="checkbox"/> Magnetização feita em múltiplas direcções. Magnetization done in multiple directions.	
<b>Não aceitável</b> Not acceptable		<input type="checkbox"/> Sample n°1 had 3,04 mm lenght cracks Sample n°2 had 2,52 mm lenght cracks Sample n°3 had 2,20 mm lenght cracks Sample n°4 had 2,86 mm lenght cracks	
<b>Aceitável com registos</b> Acceptable with notes		<input checked="" type="checkbox"/>	
<b>OPERADOR</b> TECHNICIAN		<b>RESPONSÁVEL</b> RESPONSIBLE	
<b>NOME/NAME:</b> Paulo Jorge Lima		<b>NOME/NAME:</b> Acácio Lima	
<b>Nº CERT./CERT. NO:</b> END 582		<b>Nº CERT./CERT. NO:</b> END 373	
<b>CARGO/POSITION:</b> Level II		<b>CARGO/POSITION:</b> Level III	
<b>RÚBRICA E CARIMBO/SIGN&amp;STAMP:</b>		<b>RÚBRICA E CARIMBO/SIGN&amp;STAMP:</b>	
		<b>DATAS</b> DATES	
		<b>INÍCIO/BELOWING:</b> 01 de Julho de 2014	
		<b>FIM/CLOSURE:</b> 01 de Julho de 2014	
		<b>VERIFICAÇÃO/VERIFICATION:</b> 03 de Julho de 2014	

Mod.PQ30.00/07

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Página/Page: 1 de 1

## 16. Radiographic Test Report after SCC test

 <b>A. JORGELIMA</b> <small>TESTING LABORATORY</small>		<b>RELATÓRIO RADIOGRÁFICO (DETECTORES DIGITAIS)</b> <i>RADIOGRAPHIC REPORT (DIGITAL DETECTORS)</i>				
Cliente: Client:	Traterme, Lda - Travessa do Campo da Telheira, 211 - Vila Nova da Telha	Encomenda nº: Order no:	OS 272 (Ordem de Serviço 2014)			
Relatório nº: Report no:	RT-14-295	Data e local de execução: Date and place:	01 de Julho de 2014 Rua de Serralves nº 328			
Propósito da inspeção: Inspection purpose:	Det. de descontinuidades Discontinuities detection	Fonte: Source:	Golden Engineering - XRS3			
Identificação peça: Part identification:	Sample 1 to 4	Tensão/Actividade: Tension/Activity:	270 kVp			
Desenhos aplicáveis: Drawing no:	---	Ponto focal: Focal dimension:	3,0 x 3,0 mm			
Tipo de peça: Part type:	Soldada Welded	Distância focal (mm): Film-Focus distance (mm):	600 mm			
Tipo de junta (se aplicável): Weld joint (if applicable):	Topo Butt	IQI:	EN 462-1 FeW13			
Material: Material:	P91	Ampliação: Magnification:	1x			
Espessura do material: Material thickness:	6 mm	SRa:	0,127 mm			
Tamanho de Pixel: Pixel size:	127 µm	SNR <sub>s</sub> :	92,65			
Processo de Calibração: Calibration process:	Averaging (3x)	Ganho: Gain:	1x			
Instruções e normas de ensaio: Instruction and testing standards:	EN ISO 17636-2: 2013		<b>Técnica /Technique</b> 			
Critério de aceitação: Evaluation references:	EN 12517 - Nível 2					
Filtros digitais: Digital filters:	High Pass Filter					
Detector digital: Digital detector:	Vidisco RayzorX Pro 9"x8,5"					
Écrans: Screens:	---	Software:	XBit Pro V3.0.6.0.			
Número Film No	Referência References	Interpretação Interpretation	Decisão * Decision	Técnica Technique	Fio Wire	Observações Remarks
1	Sample 1	100	+	1	15	---
2	Sample 2	-	-	1	15	---
3	Sample 3	-	-	1	15	---
4	Sample 4	-	-	1	15	---
<b>* Decisão:</b> <b>Conclusion:</b> Bom/Good: = Aceitável/Acceptable: / Reparar/To repair: + Para análise/To client analysis: ?						
<b>OPERADOR</b> <b>TECHNICIAN</b>  NOME/NAME: PAULO PEREIRA Nº CERTIFICADO/CERT. NO: END 490 CARGO/POSITION: II RÚBRICA E CARIMBO/SIGN&STAMP:		<b>RESPONSÁVEL</b> <b>RESPONSIBLE</b>  NOME/NAME: ACÁCIO LIMA Nº CERTIFICADO/CERT. NO: END 11 CARGO/POSITION: III RÚBRICA E CARIMBO/SIGN&STAMP:		<b>DATAS</b> <b>DATES</b> Início/Beginning: 01-07-2014 Fim/Closure: 01-07-2014 Verificação/Verification: 03-07-2014		
Mod.PQ10.15/03						